

PCT COOPERATION TREATY

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

United States Patent and Trademark
Office
(Box PCT)
Crystal Plaza 2
Washington, DC 20231
ÉTATS-UNIS D'AMÉRIQUE

in its capacity as elected Office

Date of mailing: 08 July 1999 (08.07.99)	
International application No.: PCT/IB98/01039	Applicant's or agent's file reference: PCT-03/98
International filing date: 07 July 1998 (07.07.98)	Priority date: 23 December 1997 (23.12.97)
Applicant: DARMAWAN, Arianto	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International preliminary Examining Authority on:
15 May 1999 (15.05.99)

☐ in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was

☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No.: (41-22) 740.14.35	Authorized officer: J. Zahra Telephone No.: (41-22) 338.83.38
---	---

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference PCT-03/98	FOR FURTHER ACTION see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. PCT/IB 98/ 01039	International filing date (day/month/year) 07/07/1998	(Earliest) Priority Date (day/month/year) 23/12/1997
Applicant DARMAWAN, Arianto		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 2 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. ☐ **Certain claims were found unsearchable** (see Box I).
2. ☐ **Unity of invention is lacking** (see Box II).
3. ☐ The international application contains disclosure of a **nucleotide and/or amino acid sequence listing** and the international search was carried out on the basis of the sequence listing
 - ☐ filed with the international application.
 - ☐ furnished by the applicant separately from the international application.
 - ☐ but not accompanied by a statement to the effect that it did not include matter going beyond the disclosure in the international application as filed.
 - ☐ Transcribed by this Authority
4. With regard to the **title**,
 - ☐ the text is approved as submitted by the applicant
 - ☒ the text has been established by this Authority to read as follows:
ION EXCHANGE SYSTEM USING U-TUBE PRINCIPLE
5. With regard to the **abstract**,
 - ☒ the text is approved as submitted by the applicant
 - ☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this International Search Report, submit comments to this Authority.
6. The figure of the **drawings** to be published with the abstract is:
 - Figure No. ☐ as suggested by the applicant. ☐ None of the figures.
 - ☐ because the applicant failed to suggest a figure.
 - ☐ because this figure better characterizes the invention.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/IB 98/01039

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 1688915	A	23-10-1928	NONE	
GB 1068403	A		NONE	
DE 1642849	A	29-04-1971	NONE	

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/IB98/01039

15.2.2000 introduce subject-matter which clearly extends beyond the content of the application as filed, contrary to Article 34(2)(b) PCT.

Concerning dependent claim 3, inlet ports located at the bottom of the U-type tube or a backwash operation using these inlet ports are not disclosed anywhere in the original description, original figures or original claims.

Concerning independent claim 4 as well as its dependent claim 5, an ion exchange process using a modified U-tube of 180° angle, which is a vertical straight tube, having backwash ports located above the lower nozzle bed or under the above nozzle bed is not disclosed in the original description, original figures or original claims.

These claims, the figure and the paragraphs on page 13 have to be deleted. Since they introduce completely new subject-matter they have not been examined with regard to novelty, inventive step and industrial applicability.

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Prior art

D1 (US-A-1 688 915) is considered to represent the closest prior art. It discloses an ion exchange process using U-tube principle consisting of two connected compartments including a service operation, a regeneration and a backwash step (figure 2, page 1, line 96- page 2, line 75). The object of D1 is to provide an ion exchange process in which no screens are required for supplying the resin.

D2 (GB-A-1 068 403) and D3 (DE-A-1 642 849) disclose ion exchange processes using U-tube principle. They do not disclose the use of separate backwash ports to remove broken resin particles of sizes larger than the nozzle openings.

Novelty

Although independent claim 1 is not clear (see item VIII), it seems to differ from D1 in that the backwashing utilizes controllable backwashing ports located below

the nozzle bed whereas in D1 they are no separate backwash ports at all: service liquid, regeneration liquid and backwash liquid all go through the same nozzles.

Inventive Step

The problem to be solved is to propose a process with an efficient backwash step if the pressure drop is too high.

The solution is to introduce separated backwash ports below the nozzle bed in order to remove broken resin particles larger than the nozzle openings. Efficient backwashing can then be conducted by adjusting the backwash flow rate to fluidize the resin bed and to carry away the particles with the backwash waste.

D1 is not related to the problem of efficient backwashing and does not disclose the solution. Therefore, claims 1 and 2 seem to be inventive (see remark under item VIII).

Re Item VIII

Certain observations on the international application

The application does not meet the requirements of Article 6 PCT, because claim 1 is not clear.

Claim 1 defines an ion exchange process without defining how this process is to be carried out, with the exception of the backwashing step. The person skilled in the art is not able to understand claim 1 without having read the description. The service step and the regeneration step as well as the way to carry them out, as described for instance in claim 2, received on 15.5.1999 with letter of 21.12.1998, have to be included in claim 1 in order to fully understand the ion exchange process.

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference PCT-03.98	FOR FURTHER ACTION		See Notification of Transmittal of International Preliminary Examination Report (Form PCT/ISA 416)
International application No. PCT IB98 01039	International filing date (day month year) 07 07 1998	Priority date (day month year) 23 12 1997	
International Patent Classification (IPC) or national classification and IPC B01J47:02			
Applicant DARMAWAN, Arianto			

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.



2. This REPORT consists of a total of 6 sheets, including this cover sheet.

☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 21 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☒ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability, citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☒ Certain observations on the international application

Date of submission of the demand 15 05 1999	Date of completion of this report 21 03 2000
Name and mailing address of the international preliminary examining authority  European Patent Office D-80289 Munich Tel: +49 89 2399-1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 21, 22, 23, 24, 25, 26, 27, 28, 29, 30, 31, 32, 33, 34, 35, 36, 37, 38, 39, 40, 41, 42, 43, 44, 45, 46, 47, 48, 49, 50, 51, 52, 53, 54, 55, 56, 57, 58, 59, 60, 61, 62, 63, 64, 65, 66, 67, 68, 69, 70, 71, 72, 73, 74, 75, 76, 77, 78, 79, 80, 81, 82, 83, 84, 85, 86, 87, 88, 89, 90, 91, 92, 93, 94, 95, 96, 97, 98, 99, 100 Fax: +49 89 2399-4465	Authorized officer  Miebach, V Telephone No: +49 89 2399-4470

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/IB98/01039

I. Basis of the report

1. This report has been drawn on the basis of *(substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments)*

Description, pages:

1-18 as received on 21.02.2000 with letter of 15.02.2000

Claims, No.:

1-5 as received on 21.02/2000 with letter of 15.02.2000

Drawings, sheets:

1/12-12/12 as originally filed

2. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

III. Non-establishment of opinion with regard to novelty, inventive step and industrial applicability

The questions whether the claimed invention appears to be novel, to involve an inventive step (to be non-obvious) or to be industrially applicable have not been examined in respect of:

- ☐ the entire international application,
☒ claims Nos. 3-5

because

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/IB98/01039

- ☐ the said international application, or the said claims Nos. relate to the following subject matter which does not require an international preliminary examination (*specify*):
- ☐ the description, claims or drawings (*indicate particular elements below*) or said claims Nos. are so unclear that no meaningful opinion could be formed (*specify*):
- ☒ the claims, or said claims Nos. 3-5 are so inadequately supported by the description that no meaningful opinion could be formed.
- ☐ no international search report has been established for the said claims Nos. .

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes: Claims 1-2
	No: Claims
Inventive step (IS)	Yes: Claims 1-2
	No: Claims
Industrial applicability (IA)	Yes: Claims 1-2
	No: Claims

2. Citations and explanations

see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET

International application No. PCT/IB98/01039

Re Item I

Basis of the report

1. The letter of the applicant dated 15.02.2000 has been taken into consideration.
2. The amendments of page 1 (introductory phrase), pages 2-5 (introduction of prior art) as well as of dependent claim 2 (dependency) are allowable under Article 34 (2) b) PCT.
3. Support for claim 1 can be found in the original description on page 12, lines 5-17 and figure 4c.
4. Some of the amendments filed with the letter dated 15.02.2000 introduce subject-matter which extends beyond the content of the application as filed, contrary to Article 34(2)(b) PCT. The amendments concerned are the following:

claim 1:	impurities or lumps: the original description (page 8, lines 13+14) discloses broken resin particles of sizes larger than the nozzle opening, but no impurities or lumps. "Impurities or lumps" have to be deleted.
claims 3-5:	complete: see remarks under item III.
page 7:	figure of the U-tube: although it is clear that there is a figure missing in the original disclosure, it cannot be introduced later on. The figure has to be deleted.
page 8, line 1	dan ρ is the density of the fluid: is not disclosed in the original disclosure and has to be deleted.
page 8:	figure of the vertical straight tube: see remark under item III.
page 13, lines 8-29:	complete: see remark under item III.

Re Item III

Non-establishment of opinion with regard to novelty, inventive step and industrial applicability

1. Claims 3-5, as well as the figure of the vertical straight tube (page 8) and the paragraph of page 13, lines 8-29 (support for claims 3-5) filed with the letter dated

15.2.2000 introduce subject-matter which clearly extends beyond the content of the application as filed, contrary to Article 34(2)(b) PCT.

Concerning dependent claim 3, inlet ports located at the bottom of the U-type tube or a backwash operation using these inlet ports are not disclosed anywhere in the original description, original figures or original claims.

Concerning independent claim 4 as well as its dependent claim 5, an ion exchange process using a modified U-tube of 180° angle, which is a vertical straight tube, having backwash ports located above the lower nozzle bed or under the above nozzle bed is not disclosed in the original description, original figures or original claims.

These claims, the figure and the paragraphs on page 13 have to be deleted. Since they introduce completely new subject-matter they have not been examined with regard to novelty, inventive step and industrial applicability.

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Prior art

D1 (US-A-1 688 915) is considered to represent the closest prior art. It discloses an ion exchange process using U-tube principle consisting of two connected compartments including a service operation, a regeneration and a backwash step (figure 2, page 1, line 96- page 2, line 75). The object of D1 is to provide an ion exchange process in which no screens are required for supplying the resin.

D2 (GB-A-1 068 403) and D3 (DE-A-1 642 849) disclose ion exchange processes using U-tube principle. They do not disclose the use of separate backwash ports to remove broken resin particles of sizes larger than the nozzle openings.

Novelty

Although independent claim 1 is not clear (see item VIII), it seems to differ from D1 in that the backwashing utilizes controllable backwashing ports located below

the nozzle bed whereas in D1 they are no separate backwash ports at all: service liquid, regeneration liquid and backwash liquid all go through the same nozzles.

Inventive Step

The problem to be solved is to propose a process with an efficient backwash step if the pressure drop is too high.

The solution is to introduce separated backwash ports below the nozzle bed in order to remove broken resin particles larger than the nozzle openings. Efficient backwashing can then be conducted by adjusting the backwash flow rate to fluidize the resin bed and to carry away the particles with the backwash waste.

D1 is not related to the problem of efficient backwashing and does not disclose the solution. Therefore, claims 1 and 2 seem to be inventive (see remark under item VIII).

Re Item VIII

Certain observations on the international application

The application does not meet the requirements of Article 6 PCT, because claim 1 is not clear.

Claim 1 defines an ion exchange process without defining how this process is to be carried out, with the exception of the backwashing step. The person skilled in the art is not able to understand claim 1 without having read the description. The service step and the regeneration step as well as the way to carry them out, as described for instance in claim 2, received on 15.5.1999 with letter of 21.12.1998, have to be included in claim 1 in order to fully understand the ion exchange process.

Description

AN ION EXCHANGE SYSTEM WITH COMPACTED MEDIA
DURING OPERATION AND REGENERATION
5 PROCESSES USING U-TUBE PRINCIPLE

Field of Invention

This invention is concerned with processes of liquid separation using ion exchange system and in particular forms is related to method for
10 demineralizing or softening of water, purification of aquaous solution, recovery of metals and valuable compounds.

Background of Invention

Ion exchange system is a process of liquid separation. Generally this
15 process uses ion exchange resin, adsorbent resin or catalytic resin.

Ion exchange resins are polymeric beads, granulas or powders having a functional acidic group called cation resin and basic group called anion. They work by exchanging ions in a solution with ions fixed inside the porous polymer matrix of the resin. Adsorbent resins are more porous and work by
20 attracting materials onto the large surfaces of the resin. Catalytic resins are polymer beads or powders with acidic or basic groups and provide an effective, environmentally friendly alternative to liquid catalyst.

In most cases involving ion exchange system the resin is confined in a column. The system may contain one or more columns filled with one or
25 more types of resin of the same or different function. The particular advantage of such ion exchange liquid separation systems as described above lies in their effectiveness, high efficiency, simplicity and low operating cost compared to other separation processes such as evaporation, reverse osmosis and the like. Successful liquid separation processes using
30 ion exchange system depend in major part, upon the characteristic of the system utilized. Among the desired characteristics are:

- High purity of the processed product.

The system must be able to ensure production of high quality of demineralized or adsorbed product. It should keep the resin bed in a
35 compacted form and reduce the ion leakage.

- Low chemical consumption

Regeneration of the exhausted resin should be performed efficiently for optimum chemical consumption and should maximize the resin bed operating capacity such as to produce the most highly regenerated resin at the service flow outlet.

- Less waste during regeneration

Regeneration is to be performed efficiently, less time, less water consumption for backwashing and rinsing, and less chemical waste.

- Reduced pressure drop

High flow rate increases pressure drop. High pressure drop increases possibility of breakage of the resin beads and causes higher pumping energy.

- Wide range of flow rates

Since most processes are not constant the system should be operable for processes at wide range of flow rates and is still capable to produce highly purified product.

- Low operating and investment cost

The system should be easily operable with low operating, investment and maintenance costs.

The following basic ion exchange systems are generally available.

The first system is a cocurrent system where the direction of flow during service is from top to bottom while the regeneration process is also from top to bottom (DEGREMONT. WATER TREATMENT HANDBOOK, 5th EDITION, JOHN WILEY & SONS 1979 PAGE 319-320). In this system the resin bed is compacted during service and regeneration. During service the liquid is passed through a column which contains resin beads supported by underdrain which normally contains nozzles at the bottom of the tank. There is a free board above the surface of the resin almost as high as the resin bed to allow the resin bed to be effectively backwashed before the regeneration process to clean and remove the small broken particles.

This cocurrent system is widely used since it is simple but requires plenty of regenerant. Because the regenerant flow is from top to bottom the highly regenerated resin bed is at the service flow inlet while the lower regenerated resin bed is at the service flow outlet. Therefore the purity of the processed products is not very high. This system requires a lot of regenerant and should be backwashed before each regeneration process.

The second system is a counter current system where the direction of flow during service is from top to bottom while the flow of the regeneration process is from bottom to top (THE UPCORE SYSTEM, DOW CHEMICAL COMPANY, FORM No.17701527895 CH 17230 E 0895 R). The upper part of the column is equipped with upper bed containing nozzles identical to that of the underdrain bed. There is a free board above the resin bed to accommodate 5-10 % expansion of the resin bed after being exhausted.

The disadvantage of this system is that during the regeneration process, where the regenerant flow is from bottom to top, the resin will not be fully compacted due to the tendency of fluidization which causes the regeneration process less effective. Because of the low upflow speed of the limited quantity of regenerant as well as the tendency of the resin bed to fall down due to the higher specific gravity of the resin as compared to that of the liquid, more regenerant are required during regeneration although it is still less than that of the cocurrent system.

Tank diameter is correlated to the upflow speed of the regenerant. Increasing the upflow speed would result in smaller tank diameter and smaller resin volume and therefore shorter regeneration cycle.

There is no backwashing facility and the resin should be discharged from the column if the pressure drop of the system exceeds the limit due to the accumulation of the broken resin particles on the nozzles opening. This system requires specially sized resin with more uniform particles to reduce pressure drop.

The third system is a counter current system where the direction of flow during service is from bottom to top and the regeneration process is conducted from top to bottom (AMBERPACK BACKWASHABLE PACKED BED SYSTEM [EXTERNAL BACKWASHING] ROHM AND HASS; PRINT INF 9003 A DEC 93 IMPACTES RCS B 350424636). The upper part of the

column is equipped with upper bed which contains nozzles identical to that of the underdrain bed. There is a free board above the resin bed to accommodate 5% to 10 % expansion of the resin bed after being exhausted.

5 The disadvantage of this system is that during service, where the flow is from bottom to top, the flow speed must be relatively high in order to obtain a compacted resin bed. In many operations the service flow rate is fluctuating or interrupted which causes fluidization or fall of the resin bed. This makes the resin bed uncompacted and contaminates the highly
10 regenerated resin at the flow outlet which will reduce the water quality and exchange capacity.

 Small broken resin particles tends to rise to the top and resin fines with the size smaller than the nozzle openings may be transported and contaminate the other ion exchange vessel. To protect the system a resin
15 trap should be installed, but it will increase the pressure drop. There is no internal backwashing facility and the resin should be discharged to be backwashed outside of the column if the pressure drop of the system exceeds the limit due to the accumulation of the broken resin particles having sizes larger than the nozzle openings. This system requires specially
20 sized resin with more uniform particles to reduce pressure drop.

 US Patent 1 688 915 (year: 1928) utilizes two connected compartments as apparatus for treating liquids. This invention emphasized that no screen are required for carrying the weight of the softening material because the weight of the softening material is carried directly by the tanks
25 or containers themselves. No compaction of the softening material and counter current effect was intended.

 One can understand this phenomenon since at the time this invention was filed, softening material contain mostly of minerals which are much heavier than nowadays resin. This system does not have a separate
30 backwashing port.

 The tank diameter is correlated with the upflow speed during service where increasing the speed requires smaller tank diameter and therefore yields smaller resin volume and consequently shorter regeneration cycle.

 The fourth system is a counter current system with water or air hold
35 down (COUNTER CURRENT ION EXCHANGE SYSTEMS IN INDUSTRIAL

AND UTILITY WATER TREATMENT. V.R. DAVIES. ROHM AND HAAS COMPANY, MARCH 1989). This system is a down flow service and upflow regeneration. In the water hold down system, there is a free board above the resin bed with distributor at the top. Regenerant exit distributor is buried just below the upper surface of the resin bed.

During regeneration cycle, the regenerant will flow upward from the bottom and exit through this collector while at the same time water will flow downward from the distributor through this exit distributor to maintain packed resin bed. In the air hold down system, air is used instead of water for the regeneration process.

The disadvantage of this system is that it requires a relatively large quantity of water or air during regeneration to compact the resin bed. Besides, the operation and maintenance are more complicated.

Counter current system shows advantages over cocurrent system. During regeneration process the resin bed at the regenerant inlet will be highly regenerated with the highest exchange capacity while the resin bed at the regenerant outlet will remain partially in the exhausted stage and has lower exchange capacity. In the cocurrent system where the service inlet is the same as the regenerant inlet, this partially exhausted stage will affect the leakage influencing the quality of the treated liquid. It is possible to increase the quality of the treated liquid by imposing higher regeneration level to convert the partially exhausted stage resin to become more highly regenerated but it will be less economical.

In the counter current system the outlet liquid will be in contact with highly regenerated portion of the resin during the service which yields much higher quality of processed liquid with less leakage and less regenerant cycle.

Summary of invention

The principal objective of this invention is developing a design of an ion exchange system for producing the highest purity of processed product with the following features: low chemical consumption, easy operation and maintenance, less waste, low operating cost, low investment cost and has a wide range of application.

Another objective of this invention is developing a design of a counter

current ion exchange system which can tolerate changes of flow rates during service cycle or even interruption of service flow. Furthermore the system should have the capability of internal backwashing whenever required to remove the resin fines if the pressure drop exceeds the expected limit. The system can be operated at low service flow rate with low pressure drop so that besides specially sized resin, standard sized resin may be used.

The aforementioned principal objective has been achieved by building a system operating on a counter current base where the resin is always compacted during service cycle, regenerated cycle or even when the flow is interrupted. The system is also provided with backwashing facility where the backwashing flow rate can be adjusted. The backwashing is in effect only if the pressure drop of the system increases such as to exceed a certain limit. In normal condition backwashing is required only after more than one hundred regeneration cycles, so there is practically no backwashed waste water in the regeneration process except once a year or whenever required.

The design of the system of this invention comprises a vertical column divided into two vertical compartments with free space in the lower part so that both compartments are interconnected and form a U tube type connection. They are filled with one or more types of ion exchange resin. The upper part of each compartment is equipped with upper bed containing nozzles. There is a free board above the resin bed to accommodate the expansion of the resin bed occurring after being exhausted or during regeneration. The direction of flow during service cycle is from top to bottom in one compartment and upward in the other compartment. The direction of flow during regeneration process, which is conducted from the other compartment, is also from top to bottom and then flows upward through the other side.

One configuration is a vertical column usually round in shape with vertical partition inside which divides this column into two vertical compartments with free space at the lower part so that it forms a U tube type connection.

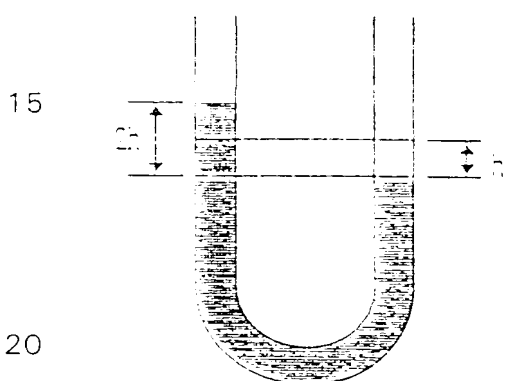
The second configuration is a vertical column usually round in shape with another vertical column located concentrically inside. There is a free

space interconnecting both columns at the bottom part so that they form a U tube type connection.

The third configuration comprises two vertical columns usually round in shape, interconnected together at the lower part to form a U tube type connection.

Because of the U tube form, the force required to compact the resin during service cycle as well as during regeneration cycle is very small. Consequently low velocity is sufficient to compact the resin. The equation is based on the drag force applied by flow against particle.

Consider the U tube model of the present invention as shown in the following figure.



ρ_p is the density of the particle (resin)

A is the cross section of the tube

u is the fluid velocity

g is the gravity constant

The fluid exerts a drag force F_d on the resin such that the resin column is displaced a distance h on each side of the tube.

Suppose the fluid flows from the right tube downward and upward to the other tube. The surface of the resin in the right tube is displaced down a distance h and in the left tube the surface of the resin is displaced up a distance h , so that the difference between the level of the resin on each tube is $2h$. The work required to move the resin column is $F_d h$. If the friction force is f_r , then the energy balance gives:

$(F_d h - f_r h) = \frac{1}{2} \rho_p A g h^2$ where the right hand side is the raise of the potential energy of the resin. Therefore

$$(F_d - f_r) = \frac{1}{2} \rho_p A g h \quad (1)$$

But the drag force is equal to

$$F_d = C_a \rho u^2 / 2g_c \quad (2)$$

where C is a constant, and ρ is the density of the fluid.

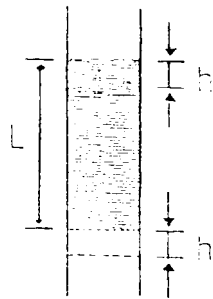
Combining Eqs.(1) and (2) yields:

$$u = \sqrt{(\rho_p g g_c h / C \rho + f_r \cdot 2 g_c / C A \rho)} \quad (3)$$

Now consider a conventional system with a resin column in a straight tube as shown below. The resin column is displaced a distance h to make it compact. Suppose the length of the resin column is L and its mass is m .

Energy balance equation yields:

$$(F_d - f_r - mgL)h = \frac{1}{2} \rho_p A g h^2 \quad (4)$$



Substituting $F_d = C A \rho u^2 / 2 g_c$ gives :

$$u = \sqrt{(\rho_p g g_c h / C \rho + f_r \cdot 2 g_c / C A \rho + 2 m g g_c L / C A \rho)} \quad (5)$$

Comparison of Eqs.(3) and (5) shows that the required velocity to make the resin compact in the conventional system is higher than that of the present invention.

It has been discovered that the resin bed remains compacted even when the cycle of the system is interrupted. This is because the resistance of the resin bed is higher than the gravitational force that would move the resin from its initial position due to the different surface level of the resin in both columns.

Compacted bed increases the performance of regeneration and service cycle. During regeneration a compacted bed will keep the resin bed firm so that the exchange of ions will be most effective with the highly regenerated zone at the regenerant inlet which is also the service outlet of the service cycle.

The system is self cleaning. During regeneration the fine broken particles which may migrate to the top of the resin bed will be carried away together with the regenerant waste. The fine particles in the other

compartment will be carried away during the rinsing process. Due to the self cleaning mechanism, in most cases no backwashing is required.

5 Nevertheless this system is equipped with internal backwashing system. After several hundreds of operation cycles broken resin particles of sizes larger than nozzle opening may accumulate which will increase the pressure drop above the acceptable limit. Backwashing can be conducted by adjusting the backwash flow rate to fluidize the resin bed and carry the fine particles away with the backwash waste.

10 Another advantage of this system compared to other cocurrent systems is that this system may use larger tank with larger volume of resin so that longer backwash interval can be obtained because this system is not affected by low service and regeneration flow rates.

Brief Description of Figures

15 The present invention will now be further described in the accompanying drawings, in which:

 Fig.1 is a schematic representation of a currently available cocurrent system (the prior art)

20 Fig.2A is a schematic representation of a currently available counter current system with down flow service and upflow regeneration (the prior art) operating in the normal down flow service mode.

 Fig.2B is a schematic representation of a currently available counter current system with down flow service and upflow regeneration (the prior art) operating in the regeneration mode.

25 Fig.3A is a schematic representation of a currently available counter current system with upflow service and down flow regeneration (the prior art) operating in the normal upflow service mode.

30 Fig.3B is a schematic representation of a currently available counter current system with upflow service and down flow regeneration (the prior art) operating in the regeneration mode.

 Fig.4A is a schematic representation of separation system according to the present invention operating in the normal service mode.

 Fig.4B is a schematic representation of separation system according to the present invention operating in the regeneration mode.

Fig.4C is a schematic representation of separation system according to the present invention operating in the backwash mode, backwashing the left column.

5 Fig.4D is a schematic representation of separation system according to the present invention operating in the backwash mode, backwashing the right column.

Fig.5 is a schematic representation of softening process according to the present invention operating in the service.

10 Fig.6 is a schematic representation of water demineralizing process according to the present invention using strong acid cationic resin for the cation column and strong base anionic resin for the anion column, operating in the service mode.

15 Fig.7 is a schematic representation of demineralizing and decolourization of sugar solution according to the present invention using strong acid cationic resin for the cation column and strong base macrorecteular anionic resin for the anion column, operating in the service mode.

20 Fig.8 is a schematic representation of water demineralizing process according to the present invention using combination of weak acid cation resin and strong acid cation resin for the cation column, degasifier, combination of weak base anion resin and strong base anion resin for the anion column with strong acid cation polisher and strong base anion polisher.

25 Fig.9A is a schematic side sectional view of one of the apparatus according to the present invention.

Fig.9B is a schematic top sectional view of one of the apparatus according to the present invention.

Fig.10A is a schematic side sectional view of one of the apparatus according to the present invention with an alternative design.

30 Fig.10B is a schematic top sectional view of one of the apparatus according to the present invention with an alternative design.

Fig.11A is a schematic side sectional view of one of the apparatus according to the present invention with another alternative design.

35 Fig.11B is a schematic top sectional view of one of the apparatus according to the present invention with another alternative design.

Complete description of invention

Figure 1 is a simplified schematic representation of a currently cocurrent system (the prior art). Column 1 with nozzle equipped underdrain bed 4 is filled with ion exchange resin about 50 % to 60 % of the total volume. Above the resin bed is freeboard 7. During service the liquid flows from port 2, which comes into contact with the compacted resin bed 5 and flows out to the outlet through port 3. During regeneration the direction of flow of the regenerant is identical to the service operation. Before the regeneration the resin is to be backwashed with the direction of flow from port 3 as the inlet to the outlet through port 2. This upflow direction will fluidize the resin bed 5, and resin fines and contaminated particles will be removed during this process. The resin level and backwash effectiveness can be monitored through sight glass 6.

Figure 2a and 2b are schematic representations of a currently available counter current system with down flow service and upflow regeneration (the prior art). Column 11 with nozzle equipped underdrain bed 14 and upper bed 15 is filled with ion exchange resin. During service (Figure 2a) the fluid is directed through port 12 passing the compacted resin bed 16a with outlet at port 13. During regeneration (Figure 2b) the regenerant passes through port 13 and the resin bed 16b with outflow through port 12. The resin will not be compacted and this will affect the regeneration efficiency. The level of the resin can be monitored from sight glass 18. Free board 17a and 17b are about 20 cm to 30 cm height just to allow the resin to expand during service operation. This system does not have an internal backwashing system. Therefore when the resin has to be backwashed it should be removed from the column to an external resin cleaning system.

Figures 3a and 3b are schematic representations of a currently available counter current system with upflow service and down flow regeneration (the prior art). Column 21 with nozzle equipped underdrain bed 24 and upper bed 25 is filled with ion exchange resin. During service (Figure 3a) the fluid flows through port 23, passes the compacted resin bed 26a and flows out through port 22. The service flow should be controlled at such a speed to maintain the resin bed in a compacted state so that the counter current regeneration profile of the resin will always be obtained. Lower speed or interrupting the flow will cause the resin not in a compacted

state and destroy the regeneration profile of the resin which will affect the quality of the treated liquid.

During regeneration (Figure 3b) the regenerant passes through port 22 and the resin bed 26b with outflow through port 23. In this cycle the resin bed is always compacted and the regeneration efficiency is high. The level of the resin can be monitored through sight glass 28. Free board 27a and 27b are of about 20 to 30 cm height to allow the resin to expand during the service operation. This system does not have an internal backwashing system. Therefore when the resin has to be backwashed, it should be removed to an external resin cleaning system.

Figures 4a, 4b, 4c and 4d are schematic representation systems according to the present invention. Column 101 is divided into two compartments by partition 110 where the lower part is interconnected such as to form a U tube type connection. Nozzle equipped upper bed 104 and 105 are located at the upper part of this column.

During service (Figure 4a) the fluid flows through port 102, passes the compacted resin 106b downflow and upflows through resin 106a with outlet at port 103. A low flow speed is sufficient to compact the resin bed and once the resin is compacted it will remain in a compacted state even if the flow speed is reduced or interrupted. Since the resin is always compacted the counter current regeneration profile will always be obtained which yields high quality of product liquid.

During regeneration (Figure 4b) the regenerant flows through port 103, passes the compacted resin 106d downflow and upflows through resin 106c with outlet at port 102. A low speed of regenerant flow is sufficient to compact the resin so that the regeneration efficiency is very high. The level of the resin can be monitored through sight glass 109. Free board 108a and 108b are of about 20 to 30 cm height just to allow the resin to expand during the service operation.

Since the system is self cleaning, backwashing to remove resin fines or contaminated particles is normally not required. If after many operation cycles the pressure drop increases such that its value becomes double, internal backwashing may be conducted. When backwashing the left column (Figure 4c), the flow is conveyed through port 103, passes the resin 106e downflow and upflows through resin 106f, then flows to the outlet through

port 111. The flow is controlled by means of flow controller 114 so that the resin will be fluidized and the fine particles can be carried away by the outlet flow. When backwashing the right column, the flow goes through port 102, passes the resin 106g downflow and upflows through resin 106h, and
5 flows out through port 112. The flow is controlled through flow controller 114 so that the resin will be fluidized and the fine particles can be carried away by the outlet flow.

Alternatively, additional backwash port may be located at the bottom of the tube for the inlet of the backwash fluid. In this mode the
10 backwashing process can be conducted for either one of the two columns or for both columns simultaneously. Backwashing the left column from the bottom inlet is conducted by closing port 112 so that the backwash fluid flows from the bottom inlet passing through the resin bed 106f and flows to the outlet through port 111. Backwashing the right column from the
15 bottom inlet can be conducted by closing port 111 so that the backwash fluid flows from the bottom inlet passing through the resin bed 106h and flows out through port 112. Backwashing both columns simultaneously is conducted by opening ports 111 and 112 and flowing the backwash fluid through the bottom inlet port passing through the resin bed 106f (or 106g) and 106e (or 106h) and outflowing through ports 111 and 112.
20

The backwashing process is also applicable for modified U-tube of 180° angle (vertical straight tube) equipped with upper and lower bed with internal backwashing utilizing controllable backwash port located above the lower nozzle bed and outlet backwash port located below the upper nozzle
25 bed in order to remove broken resin particles, impurities or lumps of sizes larger than the nozzle openings. Backwashing the resin is conducted by passing the backwash fluid with controlled flowrate through the inlet lower port located above the lower nozzle bed and outflowing through the backwash outlet port located below the upper nozzle bed.

30 For effective backwash cleaning port 111 and port 112 are to be equipped with resin trap or screen of 0.3 mm to 0.4 mm opening so that the fine particles may be driven out through this opening.

Figure 5 is a softening process of water using the system of the present invention. Tank 201 is filled with strong cation resin in sodium form
35 206a and 206b. Regenerant is sodium chlorine solution.

Figure 6 is a demineralizing process of water using the system of the present invention. Tank 301a is filled with strong acid cation resin 306ab and 306aa, while tank 301b is filled with strong base anion resin 306ba and 306bb. Regenerants for the strong acid cation resin and the strong base anion resin are hydrochloric acid solution and sodium hydroxide solution respectively.

The service flow is conveyed from port 302a, passes the strong acid cation resin 306ab downflow and upflows through cation resin 306aa, and flows out through port 303a. From this cation exchanger the decationized water flows through port 302b, passes the strong anion resin 306bb downflow, upflows through the resin 306ba and flows out through port 303b. The product is a demineralized water of high purity.

Figure 7 is a demineralizing and decolourization process of sugar solution using the system of the present invention. Tank 401a is filled with strong acid cation resin 406ab and 406aa while tank 401b is filled with strong base macrorectecular anion resin which has the capability to adsorb colour of the sugar solution.

Regenerant for the strong acid cation resin is hydrochloric acid solution while for the macrorectecular strong base anion resin the regenerant is sodium hydroxide solution.

The service flow is conveyed from port 402a, passes the strong cation resin 406ab downflow and upflows through resin 406aa, then outflows through port 403a. From this cation exchanger the decationized sugar solution flows through port 402b, passes the strong macrorectecular anion resin 406bb downflow and upflows through resin 406ba, then outflows through port 403b. The product is a demineralized and decolourized sugar solution of high purity.

Figure 8 is a demineralizing process of water containing bicarbonate salt using the system of the present invention with degasifier and polisher. Cation exchanger tank 501a is filled with weak acid cation resin 506ab on the left compartment and strong acid cation resin 506aa on the right compartment. The use of these resin combination increases the regeneration efficiency. During regeneration with hydrochloric acid solution the regenerant flow is from port 503a downflow passing the strong cation resin 506aa. After compacting the resin the regenerant waste which still contains

hydrochloric acid at lower concentration passes through and regenerates weak acid cation resin 506ab upflow and outlets through port 502a.

Degasifier 513 contains deaerator fills 515 and equipped with air blower 514 which blows the air from the lower part of the fills upward.

5 Decationized water from the cation exchanger containing bicarbonate acid passes through the upper part of the fills and the carbon dioxide gas will be removed along with the blown air.

10 Anion exchanger tank 501b is filled with weak base anion resin 506bb in the left compartment and strong base anion resin 506ba in the right compartment. The use of this resin combination increases the regeneration efficiency.

15 During regeneration with sodium hydroxide solution the regenerant flow is from 503b downflow passing the strong anion resin 506ba. After compacting this resin the regenerant waste which still contain sodium hydroxide at lower concentration passes and regenerates the weak base anion resin 506bb upflow and outlets through port 502b.

20 Cation exchanger tank 501c is filled with strong cation exchanger 506cb and 506ca while anion exchanger tank 501d is filled with strong anion exchanger 506db and 506da. These two ion exchangers function as polisher and has the same design as the system in Figure 6. The service flow of the feed water which contains bicarbonate is conducted from port 502a, passes the weak acid cation resin 506ab downflow and upflows through the strong acid cation resin 506aa with outlet port at 503a. The decationized water which contains bicarbonate acid passes degasifier 513 and gets in contact with degasifier fills 515. The CO_2 gas from the bicarbonate acid is carried away by the blown air from blower 514. The degasified decationized water is pumped by pump 516 to the anion exchanger through port 502b, passes the weak base resin 506bb downflow and upflows through the strong base anion resin 506ba, and flows out through port 503b. The demineralized water is then polished in the cation polisher, passes port 502c and flows through strong cation resin 506cb downflow and upflows through resin 506ca with outlet at port 503c. From this cation polisher the water is then conveyed to the anion polisher through port 502d, passes the strong base anion resin 506db downflow and upflows through resin 506da, and goes outlet through port 503d. The

35

treated water passing port 503b will have a high quality deionized water with conductivity up to less than $1 \mu\text{S/cm}$ and residual silica up to less than 20 PPB and after passing through the cation and anion polisher with outlet at port 503d the conductivity can be less than $0.1 \mu\text{S/cm}$ and residual silica less than 5 PPB.

Figures 9a to 11b illustrate the schematic sectional view of the apparatus using the present invention in three configurations. Figures 9a and 9b illustrate a configuration where the vertical column 101 is divided into two compartments by partition 110 with free space at the bottom part so that it forms a U tube type connection. At the upper part of the column is an upper bed with nozzles 104 and 105. Between the upper bed and the surface of the resin bed 106b is a free board 108a to accommodate the expansion of the resin bed after the resin has been exhausted, and also to accommodate resin expansion during backwash. The level of the resin bed can be observed through sight glass 109. Port 102 is an inlet of the service fluid and outlet for the regenerant fluid while port 103 is an outlet of the service fluid and inlet of the regenerant fluid. The operation can be reversed. Ports 111 and 112 are the outlets for the backwash fluid.

Figures 10a and 10b illustrate another configuration consisting of two vertical columns forming an U tube type connection. At the upper part of the columns 601a and 601b are upper beds equipped with nozzles. Between the upper bed and the surface of the resin bed 606b is a free board 608b to accommodate the expansion of the resin bed after the resin becomes exhausted as well as to accommodate the resin expansion during backwashing. The level of the resin bed can be observed through sight glass 609. Port 602 is an inlet of the service fluid and outlet of the regenerant fluid while port 603 is an outlet of the service fluid and inlet of regenerant fluid. The operation can be reversed. Ports 611 and 612 are the outlets for backwash fluid.

Figures 11a and 11b illustrate a configuration with a vertical column 701 and a smaller vertical column 701b at the inside with a connecting space at the bottom part so that the fluid as well as the resin can flow freely from one compartment to the other compartment. At the upper part of the columns is an upper bed with nozzles 704a and 704b. Between the upper bed and the surface of the resin bed 707a is a free board 708a to

accommodate resin expansion when the resin has been exhausted or during backwash. The level of the resin bed can be observed from the sight glass 709. Port 702 is an inlet of the service fluid and outlet of the regenerant fluid while port 713 is an outlet of the service fluid and inlet of the regenerant fluid. Port 703 is an alternative of port 702 which has the same function. Port 711 is the outlet for the backwash fluid. Port 712 is an alternative of port 711 which has the same function.

Example (Comparative data)

10

The chemical requirements, conductivity and silica leakage of a system according to the present invention are compared with those of a cocurrent system which are presently widely used. Each system is calculated with the same resin type, flow rate, water analysis and approximately the same resin quantity. Both systems are operating without degasifier.

15

Flow rate : 16.5 m³/h, net run 400 m³
 Water analysis (in m.eq) : HCO₃⁻ 0.334, CO₂ 0.334, SO₄⁻ 0.850, Cl⁻ 0.500, NO₃⁻ 0.039, SiO₂ 0.194, Mg⁺⁺ 0.168, Ca⁺⁺ 0.997, Na 0.552.

20

	System of the present invention	Co-current system
Resin type	6000 l Amberjet 1200 4200 l Amberlite 96 RF 4500 l Amberjet 4200	6000 l Amberjet 1200 4500 l Amberlite 96 RF 4500 Amberjet 4200
Regenerant consumption	33 kg HCl (As 100%) 36 kg NaOH (100%)	53 kg HCl (As 100%) 36 kg NaOH (100%)
Tank diameter	1000 mm	1000 mm
Effective tank height	1700 mm	3100 mm
Conductivity	1.0-3.0 μ S/cm	3.0-6.0 μ S/cm
Average end point		
Silica leakage	0.03-0.10 mg/l	0.12-0.20
Average end point		

25

30

From the data mentioned above it can be observed that the system

of the present invention has more advantages when compared to the presently used cocurrent system. The cocurrent system consume 60% more HCl regenerant, 80% more tank height, 300 % more average conductivity level and 400% more average silica leakage as compared to the system based on the present invention. The operation and construction cost of the system based on the present invention is much lower than the presently used cocurrent system while the treated water quality produced by the system of the present invention is much better than that of the cocurrent system.

10

Claims

1. An ion exchange process using U-tube principle consisting of two connected compartments with internal backwashing utilizing
5 controllable backwashing ports located below the nozzle bed in order to remove broken resin particles, impurities or lumps of sizes larger than the nozzle openings.
2. An ion exchange process according to claim 1 in which the backwash
10 process is conducted only if the pressure drop of the service flow increases significantly, backwashing the resin at the second compartment is conducted by passing the backwash fluid with controlled flowrate through the inlet port at the first compartment and outflowing through the backwash outlet port located below the
15 nozzle bed, backwashing the resin at the first compartment is conducted by passing the backwash fluid with controlled flowrate through the outlet port at the second compartment and outflowing through the backwash outlet port located below the nozzle bed.
- 20 3. An ion exchange process according to claim 1 in which the backwash process is considered only if the pressure drop of the service flow increases significantly; backwashing process is conducted by passing the backwash fluid through the inlet port located at the bottom of the U-type tube consisting of two connected compartments and
25 outflowing through the outlet ports located below the upper nozzle beds in order to remove broken resin particles, impurities or lumps of sizes larger than the nozzle openings;
backwashing the resin in the first compartment is conducted by closing the backwash port below the upper nozzle bed of the
30 second compartment and flowing the backwash fluid through the bottom inlet passing the resin bed in the first compartment and outflowing through the backwash port located below the upper nozzle bed of the first compartment; backwashing the resin in the second compartment is conducted by closing the backwash port
35 below the upper nozzle bed of the first compartment and flowing the

backwash fluid through the bottom inlet passing the resin bed in the second compartment and outflowing through the backwash port located below the upper nozzle bed of the second compartment; simultaneous backwashing of both compartments is conducted by opening both backwash ports of the first and second compartment and flowing the backwash fluid through the bottom inlet passing the resin beds in the first and second compartments and outflowing through backwash ports below the nozzle beds of the first and second compartments.

10

4. An ion exchange process using modified U-tube of 180° angle (vertical straight tube) equipped with upper and lower bed with internal backwashing utilizing a controllable backwash port located above the lower nozzle bed in order to remove broken resin particles, impurities or lumps of sizes larger than the nozzle openings.

15

5. An ion exchange process according to claim 4 in which the backwash process is conducted only if the pressure drop of the service flow increases significantly; backwashing the resin is conducted by passing the backwash fluid with controlled flowrate through the lower port located above the lower nozzle bed and outflowing through the backwash outlet port located below the upper nozzle bed.

20

Abstract

AN ION EXCHANGE SYSTEM WITH COMPACTED MEDIA
DURING OPERATION AND REGENERATION
5 PROCESSES USING U-TUBE PRINCIPLE

10 This invention is a liquid separation system using ion exchange resin, adsorbent resin and catalytic resin based on U-tube principle. It is shown that relatively low fluid velocity is sufficient to maintain the resin bed in compacted state during the operation as well as during the regeneration processes so that high quality product, low regenerant consumption, and convenient operation are obtained.

PCTWORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : B01J 47/02, B01D 15/00	A1	(11) International Publication Number: WO 99/33568 (43) International Publication Date: 8 July 1999 (08.07.99)
(21) International Application Number: PCT/IB98/01039 (22) International Filing Date: 7 July 1998 (07.07.98) (30) Priority Data: P-973953 23 December 1997 (23.12.97) ID (71)(72) Applicant and Inventor: DARMAWAN, Arianto [ID/ID]; Jl. Hegarmanah 63, Bandung 40141 (ID). (74) Agent: PRIAPANTJA, Cita, Citrawinda; Biro Oktroi Roosseno, Chase Plaza, 11th floor, Jl. Jend. Sudirman Kav. 21, Jakarta 12920 (ID).		(81) Designated States: AU, GB, US. Published <i>With international search report.</i> <i>With amended claims.</i>
(54) Title: ION EXCHANGE SYSTEM USING U-TUBE PRINCIPLE (57) Abstract This invention is a liquid separation system using ion exchange resin, adsorbent resin and catalytic resin based on U-tube principle. It is shown that relatively low fluid velocity is sufficient to maintain the resin bed in compacted state during the operation as well as during the regeneration processes so that high quality product, low regenerant consumption, and convenient operation are obtained.		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

INTERNATIONAL SEARCH REPORT

International Application No.

PCT/IB 98/01039

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 B01J47/02 B01D15/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 B01J B01D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 1 688 915 A (ASTROM) 23 October 1928 see the whole document ---	1-8
X	GB 1 068 403 A (THE PERMUTIT CO.) 10 May 1967 see the whole document ---	1-8
X	DE 16 42 849 A (METALLGESLLSCHAFT) 29 April 1971 see page 10-11; claims 1-6 -----	1-6

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

23 October 1998

Date of mailing of the international search report

30/10/1998

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx 31 651 epo nl.
Fax: (+31-70) 340-3016

Authorized officer

Wendling, J-P

Description

ION EXCHANGE SYSTEM USING U-TUBE PRINCIPLE

5

Field of Invention

This invention is concerned with processes of liquid separation using ion exchange system and in particular forms is related to Method and Apparatus for demineralizing or softening of water, purification of aquaous solution, recovery of metals and valuable compounds.

Background of Invention

Ion exchange system is a process of liquid separation. Generally this process uses ion exchange resin, adsorbent resin or catalytic resin.

Ion exchange resins are polymeric beads, granulas or powders having a functional acidic group called cation resin and basic group called anion. They work by exchanging ions in a solution with ions fixed inside the porous polymer matrix of the resin. Adsorbent resins are more porous and work by attracting materials onto the large surfaces of the resin. Catalytic resins are polymer beads or powders with acidic or basic groups and provide an effective, environmentally friendly alternative to liquid catalyst.

In most cases involving ion exchange system the resin is confined in a column. The system may contain one or more columns filled with one or more types of resin of the same or different function. The particular advantage of such ion exchange liquid separation systems as described above lies in their effectiveness, high efficiency, simplicity and low operating cost compared to other separation processes such as evaporation, reverse osmosis and the like. Successful liquid separation processes using ion exchange system depend in major part, upon the characteristic of the system utilized. Among the desired characteristics are:

- High purity of the processed product.

The system must be able to ensure production of high quality of demineralized or adsorbed product. It should keep the resin bed in a compacted form and reduce the ion leakage.

- Low chemical consumption
Regeneration of the exhausted resin should be performed efficiently for optimum chemical consumption and should maximize the resin bed operating capacity such as to produce the most highly regenerated resin at the service flow outlet.
5
 - Less waste during regeneration
Regeneration is to be performed efficiently, less time, less water consumption for backwashing and rinsing, and less chemical waste.
10
 - Reduced pressure drop
High flow rate increases pressure drop. High pressure drop increases possibility of breakage of the resin beads and causes higher pumping energy.
15
 - Wide range of flow rates
Since most processes are not constant the system should be operable for processes at wide range of flow rates and is still capable to produce highly purified product.
20
 - Low operating and investment cost
The system should be easily operable with low operating, investment and maintenance costs.
- 25 The following basic ion exchange systems are generally available.
- The first system is a cocurrent system where the direction of flow during service is from top to bottom while the regeneration process is also from top to bottom. In this system the resin bed is compacted during service and regeneration. During service the liquid is passed through a
30 column which contains resin beads supported by underdrain which normally contains nozzles at the bottom of the tank. There is a free board above the surface of the resin almost as high as the resin bed to allow the resin bed to be effectively backwashed before the regeneration process to clean and remove the small broken particles.
- 35 This cocurrent system is widely used since it is simple but requires

plenty of regenerant. Because the regenerant flow is from top to bottom the highly regenerated resin bed is at the service flow inlet while the lower regenerated resin bed is at the service flow outlet. Therefore the purity of the processed products is not very high. This system requires a lot of
5 regenerant and should be backwashed before each regeneration process.

The second system is a counter current system where the direction of flow during service is from top to bottom while the flow of the regeneration process is from bottom to top. The upper part of the column is equipped with upper bed containing nozzles identical to that of the
10 underdrain bed. There is a free board above the resin bed to accommodate 5-10 % expansion of the resin bed after being exhausted.

The disadvantage of this system is that during the regeneration process, where the regenerant flow is from bottom to top, the resin will not be fully compacted due to the tendency of fluidization which causes the
15 regeneration process less effective. Because of the low upflow speed of the limited quantity of regenerant as well as the tendency of the resin bed to fall down due to the higher specific gravity of the resin as compared to that of the liquid, more regenerant are required during regeneration although it is still less than that of the cocurrent system.

20 Tank diameter is correlated to the upflow speed of the regenerant. Increasing the upflow speed would result in smaller tank diameter and smaller resin volume and therefore shorter regeneration cycle.

There is no backwashing facility and the resin should be discharged from the column if the pressure drop of the system exceeds the limit due to
25 the accumulation of the broken resin particles on the nozzles opening. This system requires specially sized resin with more uniform particles to reduce pressure drop.

The third system is a counter current system where the direction of flow during service is from bottom to top and the regeneration process is
30 conducted from top to bottom. The upper part of the column is equipped with upper bed which contains nozzles identical to that of the underdrain bed. There is a free board above the resin bed to accommodate 5 % to 10 % expansion of the resin bed after being exhausted.

35 The disadvantage of this system is that during service, where the flow is from bottom to top, the flow speed must be relatively high in order

to obtain a compacted resin bed. In many operations the service flow rate is fluctuating or interrupted which causes fluidization or fall of the resin bed. This makes the resin bed uncompacted and contaminates the highly regenerated resin at the flow outlet which will reduce the water quality and exchange capacity.

Small broken resin particles tends to rise to the top and resin fines with the size smaller than the nozzle openings may be transported and contaminate the other ion exchange vessel. To protect the system a resin trap should be installed, but it will increase the pressure drop. There is no internal backwashing facility and the resin should be discharged to be backwashed outside of the column if the pressure drop of the system exceeds the limit due to the accumulation of the broken resin particles having sizes larger than the nozzle openings. This system requires specially sized resin with more uniform particles to reduce pressure drop.

The tank diameter is correlated with the upflow speed during service where increasing the speed requires smaller tank diameter and therefore yields smaller resin volume and consequently shorter regeneration cycle.

The fourth system is a counter current system with water or air hold down. This system is a down flow service and upflow regeneration. In the water hold down system, there is a free board above the resin bed with distributor at the top. Regenerant exit distributor is buried just below the upper surface of the resin bed.

During regeneration cycle, the regenerant will flow upward from the bottom and exit through this collector while at the same time water will flow downward from the distributor through this exit distributor to maintain packed resin bed. In the air hold down system, air is used instead of water for the regeneration process.

The disadvantage of this system is that it requires a relatively large quantity of water or air during regeneration to compact the resin bed. Besides, the operation and maintenance are more complicated.

Counter current system shows advantages over cocurrent system. During regeneration process the resin bed at the regenerant inlet will be highly regenerated with the highest exchange capacity while the resin bed at the regenerant outlet will remain partially in the exhausted stage and has lower exchange capacity. In the cocurrent system where the service inlet

is the same as the regenerant inlet, this partially exhausted stage will affect the leakage influencing the quality of the treated liquid. It is possible to increase the quality of the treated liquid by imposing higher regeneration level to convert the partially exhausted stage resin to become more highly regenerated but it will be less economical.

In the counter current system the outlet liquid will be in contact with highly regenerated portion of the resin during the service which yields much higher quality of processed liquid with less leakage and less regenerant cycle.

Summary of invention

The principal objective of this invention is developing a design of an ion exchange system for producing the highest purity of processed product with the following features: low chemical consumption, easy operation and maintenance, less waste, low operating cost, low investment cost and has a wide range of application.

Another objective of this invention is developing a design of a counter current ion exchange system which can tolerate changes of flow rates during service cycle or even interruption of service flow. Furthermore the system should have the capability of internal backwashing whenever required to remove the resin fines if the pressure drop exceeds the expected limit. The system can be operated at low service flow rate with low pressure drop so that besides specially sized resin, standard sized resin may be used.

The aforementioned principal objective has been achieved by building a system operating on a counter current base where the resin is always compacted during service cycle, regenerated cycle or even when the flow is interrupted. The system is also provided with backwashing facility where the backwashing flow rate can be adjusted. The backwashing is in effect only if the pressure drop of the system increases such as to exceed a certain limit. In normal condition backwashing is required only after more than one hundred regeneration cycles, so there is practically no backwashed waste water in the regeneration process except once a year or whenever required.

The design of the system of this invention comprises a vertical

column divided into two vertical compartments with free space in the lower part so that both compartments are interconnected and form a U tube type connection. They are filled with one or more types of ion exchange resin. The upper part of each compartment is equipped with upper bed containing
5 nozzles. There is a free board above the resin bed to accommodate the expansion of the resin bed occurring after being being exhausted or during regeneration. The direction of flow during service cycle is from top to bottom in one compartment and upward in the other compartment. The direction of flow during regeneration process, which is conducted from the other
10 compartment, is also from top to bottom and then flows upward through the other side.

One configuration is a vertical column usually round in shape with vertical partition inside which divides this column into two vertical compartments with free space at the lower part so that it forms a U tube
15 type connection.

The second configuration is a vertical column usually round in shape with another vertical column located concentrically inside. There is a free space interconnecting both columns at the bottom part so that they form a U tube type connection.

20 The third configuration comprises two vertical columns usually round in shape, interconnected together at the lower part to form a U tube type connection.

Because of the U tube form, the force required to compact the resin during service cycle as well as during regeneration cycle is very small.
25 Consequently low velocity is sufficient to compact the resin. The equation is based on the drag force applied by flow against particle.

Consider the U tube model of the present invention as shown in the following figure.

30 ρ_p is the density of the particle (resin)
 A is the cross section of the tube
 u is the fluid velocity
 g is the gravity constant

35 The fluid exerts a drag force F_d on the resin such that the resin

column is displaced a distance h on each side of the tube.

Suppose the fluid flows from the right tube downward and upward to the other tube. The surface of the resin in the right tube is displaced down a distance h and in the left tube the surface of the resin is displaced up a distance h , so that the difference between the level of the resin on each tube is $2h$. The work required to move the resin column is $F_d h$. If the friction force is f_r , then the energy balance gives:

$(F_d h - f_r h) = \frac{1}{2} \rho_p Agh^2$ where the right hand side is the raise of the potential energy of the resin. Therefore

$$(F_d - f_r) = \frac{1}{2} \rho_p Agh \quad (1)$$

But the drag force is equal to

$$F_d = CA\rho u^2/2g_c \quad (2)$$

where C is a constant.

Combining Eqs.(1) and (2) yields:

$$u = \sqrt{(\rho_p g g_c h / C\rho + f_r \cdot 2g_c / CA\rho)} \quad (3)$$

Now consider a conventional system with a resin column in a straight tube as shown below. The resin column is displaced a distance h to make it compact. Suppose the length of the resin column is L and its mass is m .

Energy balance equation yields:

$$(F_d - f_r - mgL)h = \frac{1}{2} \rho_p Agh^2 \quad (4)$$

Substituting $F_d = CA\rho u^2/2g_c$ gives :

$$u = \sqrt{(\rho_p g g_c h / C\rho + f_r \cdot 2g_c / CA\rho + 2mg g_c L / CA\rho)} \quad (5)$$

Comparison of Eqs.(3) and (5) shows that the required velocity to make the resin compact in the conventional system is higher than that of the present invention.

It has been discovered that the resin bed remains compacted even when the cycle of the system is interrupted. This is because the resistance of the resin bed is higher than the gravitational force that would move the resin from its initial position due to the different surface level of the resin in

both columns.

Compacted bed increases the performance of regeneration and service cycle. During regeneration a compacted bed will keep the resin bed firm so that the exchange of ions will be most effective with the highly regenerated zone at the regenerant inlet which is also the service outlet of the service cycle.

The system is self cleaning. During regeneration the fine broken particles which may migrate to the top of the resin bed will be carried away together with the regenerant waste. The fine particles in the other compartment will be carried away during the rinsing process. Due to the self cleaning mechanism, in most cases no backwashing is required.

Nevertheless this system is equipped with internal backwashing system. After several hundreds of operation cycles broken resin particles of sizes larger than nozzle opening may accumulate which will increase the pressure drop above the acceptable limit. Backwashing can be conducted by adjusting the backwash flow rate to fluidize the resin bed and carry the fine particles away with the backwash waste.

Another advantage of this system compared to other cocurrent systems is that this system may use larger tank with larger volume of resin so that longer backwash interval can be obtained because this system is not affected by low service and regeneration flow rates.

Brief Description of Figures

The present invention will now be further described in the accompanying drawings, in which:

Fig.1 is a schematic representation of a currently available cocurrent system (the prior art)

Fig.2A is a schematic representation of a currently available counter current system with down flow service and upflow regeneration (the prior art) operating in the normal down flow service mode.

Fig.2B is a schematic representation of a currently available counter current system with down flow service and upflow regeneration (the prior art) operating in the backwash mode.

Fig.3A is a schematic representation of a currently available counter

current system with upflow service and down flow regeneration (the prior art) operating in the normal upflow service mode.

Fig.3B is a schematic representation of a currently available counter current system with upflow service and down flow regeneration (the prior art) operating in the backwash mode.

Fig.4A is a schematic representation of separation system according to the present invention operating in the normal service mode.

Fig.4B is a schematic representation of separation system according to the present invention operating in the regeneration mode.

Fig.4C is a schematic representation of separation system according to the present invention operating in the backwash mode, backwashing the left column.

Fig.4D is a schematic representation of separation system according to the present invention operating in the backwash mode, backwashing the right column.

Fig.5 is a schematic representation of softening process according to the present invention operating in the service.

Fig.6 is a schematic representation of water demineralizing process according to the present invention using strong acid cationic resin for the cation column and strong base anionic resin for the anion column, operating in the service mode.

Fig.7 is a schematic representation of demineralizing and decolourization of sugar solution according to the present invention using strong acid cationic resin for the cation column and strong base macrorecticular anionic resin for the anion column, operating in the service mode.

Fig.8 is a schematic representation of water demineralizing process according to the present invention using combination of weak acid cation resin and strong acid cation resin for the cation column, degasifier, combination of weak base anion resin and strong base anion resin for the anion column with strong acid cation polisher and strong base anion polisher.

Fig.9A is a schematic side sectional view of one of the apparatus according to the present invention.

Fig.9B is a schematic top sectional view of one of the apparatus

according to the present invention.

Fig.10A is a schematic side sectional view of one of the apparatus according to the present invention with an alternative design.

Fig.10B is a schematic top sectional view of one of the apparatus
5 according to the present invention with an alternative design.

Fig.11A is a schematic side sectional view of one of the apparatus according to the present invention with another alternative design.

Fig.11B is a schematic top sectional view of one of the apparatus according to the present invention with another alternative design.

10

Complete description of invention

Figure 1 is a simplified schematic representation of a currently cocurrent system (the prior art). Column 1 with nozzle equipped underdrain bed 4 is filled with ion exchange resin about 50 % to 60 % of the total
15 volume. Above the resin bed is freeboard 7. During service the liquid flows from port 2, which comes into contact with the compacted resin bed 5 and flows out to the outlet through port 3. During regeneration the direction of flow of the regenerant is identical to the service operation. Before the regeneration the resin is to be backwashed with the direction of flow from
20 port 3 as the inlet to the outlet through port 2. This upflow direction will fluidize the resin bed 5, and resin fines and contaminated particles will be removed during this process. The resin level and backwash effectiveness can be monitored through sight glass 6.

Figure 2a and 2b are schematic representations of a currently
25 available counter current system with down flow service and upflow regeneration (the prior art). Column 11 with nozzle equipped underdrain bed 14 and upper bed 15 is filled with ion exchange resin. During service (Figure 2a) the fluid is directed through port 12 passing the compacted resin bed 16a with outlet at port 13. During regeneration (Figure 2b) the regenerant
30 passes through port 13 with outflow through port 12. The resin will not be compacted and this will affect the regeneration efficiency. The level of the resin can be monitored from sight glass 18. Free board 17a and 17b are about 20 cm to 30 cm height just to allow the resin to expand during service operation. This system does not have an internal backwashing
35 system. Therefore when the resin has to be backwashed it should be

removed from the column to an external resin cleaning system.

Figures 3a and 3b are schematic representations of a currently available counter current system with upflow service and down flow regeneration (the prior art). Column 21 with nozzle equipped underdrain bed
5 24 and upper bed 25 is filled with ion exchange resin. During service (Figure 3a) the fluid flows through port 23, passes the compacted resin bed 26a and flows out through port 22. The service flow should be controlled at such a speed to maintain the resin bed in a compacted state so that the counter current regeneration profile of the resin will always be obtained.
10 Lower speed or interrupting the flow will cause the resin not in a compacted state and destroy the regeneration profile of the resin which will affect the quality of the treated liquid.

During regeneration (Figure 3b) the regenerant passes through port 22 with outflow through port 22. In this cycle the resin bed is always
15 compacted and the regeneration efficiency is high. The level of the resin can be monitored through sight glass 28. Free board 27a and 27b are of about 20 to 30 cm height to allow the resin to expand during the service operation. This system does not have an internal backwashing system. Therefore when the resin has to be backwashed, it should be removed to
20 an external resin cleaning system.

Figures 4a, 4b, 4c and 4d are schematic representation systems according to the present invention. Column 101 is divided into two compartments by partition 110 where the lower part is interconnected such as to form a U tube type connection. Nozzle equipped upper bed 104 and
25 105 are located at the upper part of this column.

During service (Figure 4a) the fluid flows through port 102, passes the compacted resin 106b downflow and upflows through resin 106a with outlet at port 103. A low flow speed is sufficient to compact the resin bed and once the resin is compacted it will remain in a compacted state even if
30 the flow speed is reduced or interrupted. Since the resin is always compacted the counter current regeneration profile will always be obtained which yields high quality of product liquid.

During regeneration (Figure 4b) the regenerant flows through port 103, passes the compacted resin 106d downflow and upflows through
35 resin 106c with outlet at port 102. A low speed of regenerant flow is

sufficient to compact the resin so that the regeneration efficiency is very high. The level of the resin can be monitored through sight glass 109. Free board 108a and 108b are of about 20 to 30 cm height just to allow the resin to expand during the service operation.

5 Since the system is self cleaning, backwashing to remove resin fines or contaminated particles is normally not required. If after many operation cycles the pressure drop increases such that its value becomes double, internal backwashing may be conducted. When backwashing the left column (Figure 4c), the flow is conveyed through port 103, passes the resin 106e
10 downflow and upflows through resin 106f, then flows to the outlet through port 111. The flow is controlled by means of flow controller 114 so that the resin will be fluidized and the fine particles can be carried away by the outlet flow. When backwashing the right column, the flow goes through port 102, passes the resin 106g downflow and upflows through resin 106h, and
15 flows out through port 112. The flow is controlled through flow controller 114 so that the resin will be fluidized and the fine particles can be carried away by the outlet flow.

For effective backwash cleaning port 111 and port 112 are to be equipped with resin trap or screen of 0.3 mm to 0.4 mm opening so that
20 the fine particles may be driven out through this opening.

Figure 5 is a softening process of water using the system of the present invention. Tank 201 is filled with strong cation resin in sodium form 206a and 206b. Regenerant is sodium chlorine solution.

Figure 6 is a demineralizing process of water using the system of the
25 present invention. Tank 301a is filled with strong acid cation resin 306ab and 306aa, while tank 301b is filled with strong base anion resin 306ba and 306bb. Regenerants for the strong acid cation resin and the strong base anion resin are hydrochloric acid solution and sodium hydroxide solution respectively.

30 The service flow is conveyed from port 302a, passes the strong acid cation resin 306ab downflow and upflows through cation resin 306aa, and flows out through port 303a. From this cation exchanger the decationized water flows through port 302a, passes the strong anion resin 306bb downflow, upflows through the resin 306ba and flows out through port
35 303b. The product is a demineralized water of high purity.

Figure 7 is a demineralizing and decolourization process of sugar solution using the system of the present invention. Tank 401a is filled with strong acid cation resin 406ab and 406aa while tank 401b is filled with strong base macrorectecular anion resin which has the capability to adsorb colour of the sugar solution.

Regenerant for the strong acid cation resin is hydrochloric acid solution while for the macrorectecular strong base anion resin the regenerant is sodium hydroxide solution.

The service flow is conveyed from port 402a, passes the strong cation resin 406ab downflow and upflows through resin 406aa, then outflows through port 403a. From this cation exchanger the decationized sugar solution flows through port 402b, passes the strong macrorectecular anion resin 406bb downflow and upflows through resin 406ba, then outflows through port 403b. The product is a demineralized and decolourized sugar solution of high purity.

Figure 8 is a demineralizing process of water containing bicarbonate salt using the system of the present invention with degasifier and polisher. Cation exchanger tank 501a is filled with weak acid cation resin 506ab on the left compartment and strong acid cation resin 506aa on the right compartment. The use of these resin combination increases the regeneration efficiency. During regeneration with hydrochloric acid solution the regenerant flow is from port 503a downflow passing the strong cation resin 506aa. After compacting the resin the regenerant waste which still contains hydrochloric acid at lower concentration passes through and regenerates weak acid cation resin 506ab upflow and outlets through port 502a.

Degasifier 513 contains deaerator fills 515 and equipped with air blower 514 which blows the air from the lower part of the fills upward.

Decationized water from the cation exchanger containing bicarbonate acid passes through the upper part of the fills and the carbon dioxide gas will be removed along with the blown air.

Anion exchanger tank 501b is filled with weak base anion resin 506bb in the left compartment and strong base anion resin 506ba in the right compartment. The use of this resin combination increases the regeneration efficiency.

During regeneration with sodium hydroxide solution the regenerant

flow is from 503b downflow passing the strong anion resin 506ba. After compacting this resin the regenerant waste which still contain sodium hydroxide at lower concentration passes and regenerates the weak base anion resin 506bb upflow and outlets through port 502b.

5 Cation exchanger tank 501c is filled with strong cation exchanger 506cb and 506ca while anion exchanger tank 501d is filled with strong anion exchanger 506db and 506da. These two ion exchangers function as polisher and has the same design as the system in Figure 6. The service
10 flow of the feed water which contains bicarbonate is conducted from port 502a, passes the weak acid cation resin 506ab downflow and upflows through the strong acid cation resin 506aa with outlet port at 503a. The decationized water which contains bicarbonate acid passes degasifier 513 and gets in contact with degasifier fills 515. The CO₂ gas from the bicarbonate acid is carried away by the blown air from blower 514. The
15 degasified decationized water is pumped by pump 516 to the anion exchanger through port 502b, passes the weak base resin 506bb downflow and upflows through the strong base anion resin 506ba, and flows out through port 503b. The demineralized water is then polished in the cation polisher, passes port 502c and flows through strong cation resin
20 506cb downflow and upflows through resin 506ca with outlet at port 503c. From this cation polisher the water is then conveyed to the anion polisher through port 502c, passes the strong base anion resin 506db downflow and upflows through resin 506da, and goes outlet through port 503d. The treated water passing port 503b will have a high quality deionized water
25 with conductivity up to less than 1 μ S/cm and residual silica up to less than 20 PPB and after passing through the cation and anion polisher with outlet at port 503d the conductivity can be less than 0.1 μ S/cm and residual silica less than 5 PPB.

30 Figures 9a to 11b illustrate the schematic sectional view of the apparatus using the present invention in three configurations. Figures 9a and 9b illustrate a configuration where the vertical column 101 is divided into two compartments by partition 110 with free space at the bottom part so that it forms a U tube type connection. At the upper part of the column is an upper bed with nozzles 104 and 105. Between the upper bed and the
35 surface of the resin bed 106b is a free board 108a to accommodate the

expansion of the resin bed after the resin has been exhausted, and also to accommodate resin expansion during backwash. The level of the resin bed can be observed through sight glass 109. Port 102 is an inlet of the service fluid and outlet for the regenerant fluid while port 103 is an outlet of the service fluid and inlet of the regenerant fluid. The operation can be reversed. Ports 111 and 112 are the outlets for the backwash fluid.

Figures 10a and 10b illustrate another configuration consisting of two vertical columns forming an U tube type connection. At the upper part of the columns 601a and 601b are upper beds equipped with nozzles. Between the upper bed and the surface of the resin bed 606b is a free board 608b to accommodate the expansion of the resin bed after the resin becomes exhausted as well as to accommodate the resin expansion during backwashing. The level of the resin bed can be observed through sight glass 609. Port 602 is an inlet of the service fluid and outlet of the regenerant fluid while port 603 is an outlet of the service fluid and inlet of regenerant fluid. The operation can be reversed. Ports 611 and 612 are the outlets for backwash fluid.

Figures 11a and 11b illustrate a configuration with a vertical column 701 and a smaller vertical column 701b at the inside with a connecting space at the bottom part so that the fluid as well as the resin can flow freely from one compartment to the other compartment. At the upper part of the columns is an upper bed with nozzles 704a and 704b. Between the upper bed and the surface of the resin bed 707a is a free board 708a to accommodate resin expansion when the resin has been exhausted or during backwash. The level of the resin bed can be observed from the sight glass 709. Port 702 is an inlet of the service fluid and outlet of the regenerant fluid while port 713 is an outlet of the service fluid and inlet of the regenerant fluid. Port 703 is an alternative of port 702 which has the same function. Port 711 is the outlet for the backwash fluid. Port 712 is an alternative of port 711 which has the same function.

Example (Comparative data)

The chemical requirements, conductivity and silica leakage of a system according to the present invention are compared with those of a

concurrent system which are presently widely used. Each system is calculated with the same resin type, flow rate, water analysis and approximately the same resin quantity. Both systems are operating without degasifier.

5 Flow rate : 16.5 m³/h, net run 400 m³
Water analysis (in m.eq) : HCO₃⁻ 0.334, CO₂ 0.334, SO₄⁻
0.850, Cl⁻ 0.500, NO₃⁻
0.039, SiO₂ 0.194, Mg⁺⁺
0.168, Ca⁺⁺ 0.997, Na 0.552.

10

	System of the present invention	Co-current system
Resin type	6000 I Amberjet 1200 4200 I Amberlite 96 RF 4500 I Amberjet 4200	6000 I Amberjet 1200 4500 I Amberlite 96 RF 4500 Amberjet 4200
Regenerant consumption	33 kg HCl (As 100%) 36 kg NaOH (100%)	53 kg HCl (As 100%) 36 kg NaOH (100%)
15 Tank diameter	1000 mm	1000 mm
Effective tank height	1700 mm	3100 mm
Conductivity	1.0/3.0 $\mu\text{S/cm}$	3.0/6.0 $\mu\text{S/cm}$
Average/end point		
Silica leakage	0.03/0.10 mg/l	0.12/0.20
20 Average/end point		

From the data mentioned above it can be observed that the system of the present invention has more advantages when compared to the presently used cocurrent system. The cocurrent system consume 60% more HCl regenerant, 80% more tank height, 300 % more average conductivity level and 400% more average silica leakage as compared to the system based on the present invention. The operation and construction cost of the system based on the present invention is much lower than the presently used cocurrent system while the treated water quality produced by the system of the present invention is much better than that of the cocurrent system.

Claims

1. An apparatus for conducting liquid separation comprising of :
5 one or more separation columns with two compartments connected one to another, in which columns have two compartments with an open space at the lower part connecting both compartments together, each separation column has inlet means and outlet means for process liquid, inlet means and outlet means for regeneration liquid, inlet means and outlet means for rinse liquid, and inlet means and outlet means for backwash liquid;
10 a plurality of valved pipings connected together to form a loop for introducing the liquid in both forward and reverse flow direction to the said separation column;
granular particles filled into the said separation column; and
15 means to feed the process liquid into the said separation column and to take off the treated liquid from the said separation column, to feed the regenerant liquid into the said separation column and to take off the regeneration waste liquid from the said separation column, to feed the rinse liquid into the said separation column and to take off the rinse waste liquid from the said separation column,
20 and to feed the backwash liquid into the said separation column and to take off the waste backwash liquid from the said separation column.
- 25 2. An apparatus as in claim 1 wherein the separation column(s) is :
a round vertical column divided by a vertical partition to form two vertical compartments with an open space at the bottom of the said column so that both compartments are connected one to another, the upperpart of each compartment has an upperbed equipped with
30 distribution nozzles, characterized in that :
- two round vertical columns forming two compartments where both of the said compartments are connected together at the lower part through an open space between the said columns, wherein the upper part of each column has an upperbed
35 equipped with distribution nozzles; and

- 5 - a round vertical collumn divided concentrically by an inside
smaller round vertical collumn with a free space at the bottom
of both said collumns so that both of the said collumns are
connected together, wherein the upper part of each collumn
has an upperbed equipped with distribution nozzles.
- 10 3. An apparatus as in claim 1 and 2 wherein the position of the collumn
is of any angle from 0° to 180° in which 360° means both collumns
form a circular configuration.
- 15 4. An apparatus as in claims 1 and 2 wherein both collumns form a
circular connection with a free space in between, the upperpart of
each collumn has an upperbed equipped with distribution nozzles.
- 20 5. An apparatus as in claims 1,2,3,and 4 wherein the cross section of
the collumn is of any other shape.
- 25 6. An apparatus as in claims 1 to 5 wherein the said granular particles
are:
 ion exchange resin, catalytic resin, adsorbent resin, inert resin
or mixture of one or more of those resin; and
 granular particles for liquid separation which have the
capability to adsorb physically or react chemically.
- 30 7. A method of liquid separation utilizing apparatus as claimed in claims
1 to 6.
- 35 8. A method of liquid separation as claimed in claim 7 comprising of :
 service operation conducted by passing the feed liquid
downflow through the feed inlet port located at the top of the first
compartment and then upflow through the second compartment with
outflow through the outlet port located at the top, the service flow
makes the resin being compacted at the first compartment with a
free board located between the upper nozzle bed and the surface of
the resin bed;

5 regeneration operation conducted by passing the regenerant liquid downflow through the outlet port at the second compartment and then upflow through the first compartment with outflow through the inlet port located at the top, the regenerant flow makes the resin being compacted with a free board located between the upper nozzle bed and the second compartment;

10 backwash operation conducted only if the pressure drop of the service flow increases significantly, backwashing the resin at the second compartment is conducted by passing the backwash fluid with controlled flowrate through the inlet port at the first compartment and outflowing through the backwash outlet port located below the nozzle bed, backwashing the resin at the first
15 compartment is conducted by passing the backwash fluid with controlled flowrate through the outlet port at the second compartment and outflowing through the backwash outlet port located below the nozzle bed.

20

AMENDED CLAIMS

[received by the International Bureau on 21 December 1998 (21.12.98);
original claims 1-8 replaced by amended claims 1-2 (1 page)]

1. An ion exchange system for the treatment of liquid in which ion
exchanger is compacted by flowing the liquid through two connected
5 compartment using the U-tube principle.
2. The compaction process of ion exchanger for the treatment of liquid
according to claim 1, comprising of:
 - 10 (a) service operation conducted by passing the feed liquid
downflow through the feed inlet port located at the top of the
first compartment and then upflow through the second
compartment with outflow through the outlet port located at
the top, the service flow makes the resin being compacted at
15 the first compartment with a free board located between the
upper nozzle bed and the surface of the resin bed;
 - (b) regeneration operation conducted by passing the regenerant
liquid downflow through the outlet port at the second
compartment and then upflow through the first compartment
20 with outflow through the inlet port located at the top, the
regenerant flow makes the resin being compacted with a free
board located between the upper nozzle bed and the second
compartment;
 - 25 (c) backwash operation conducted only if the pressure drop of the
service flow increases significantly, backwashing the resin at
the second compartment is conducted by passing the
backwash fluid with controlled flowrate through the inlet port
at the first compartment and outflowing through the backwash
30 outlet port located below the nozzle bed, backwashing the
resin at the first compartment is conducted by passing the
backwash fluid with controlled flowrate through the outlet port
at the second compartment and outflowing through the
backwash outlet port located below the nozzle bed.

1/12

Fig.1

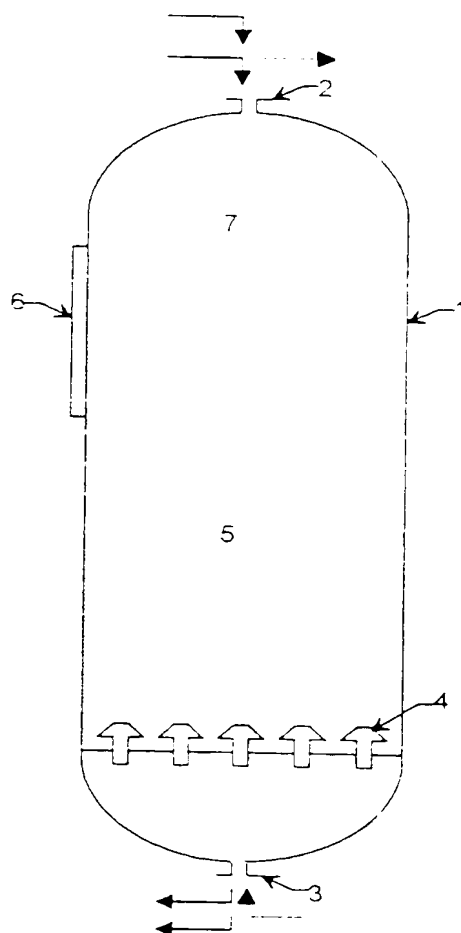


Fig.2b

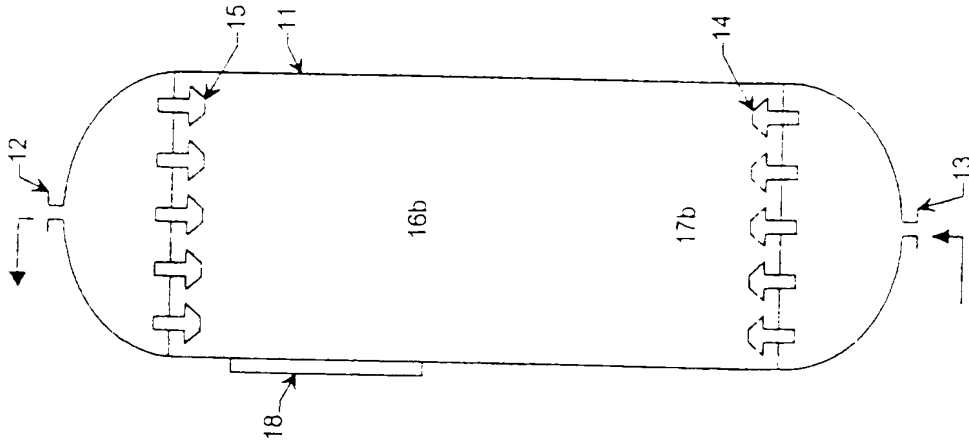


Fig.2a

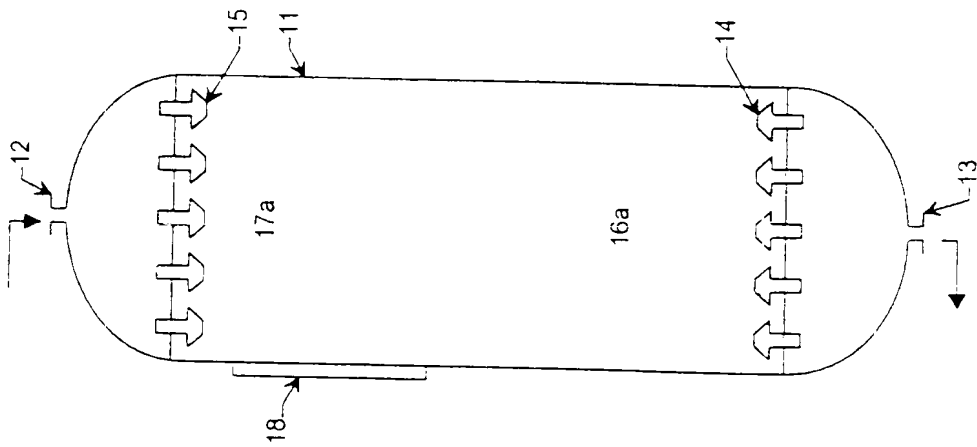


Fig. 3a

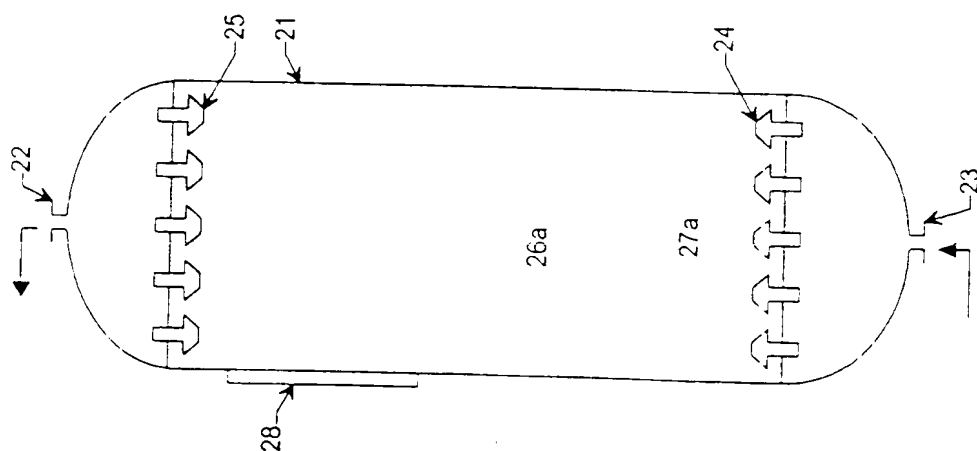
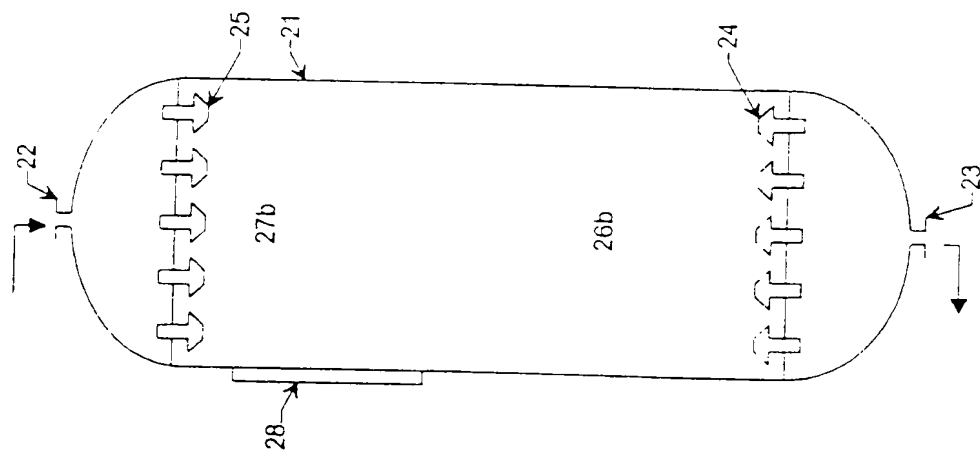
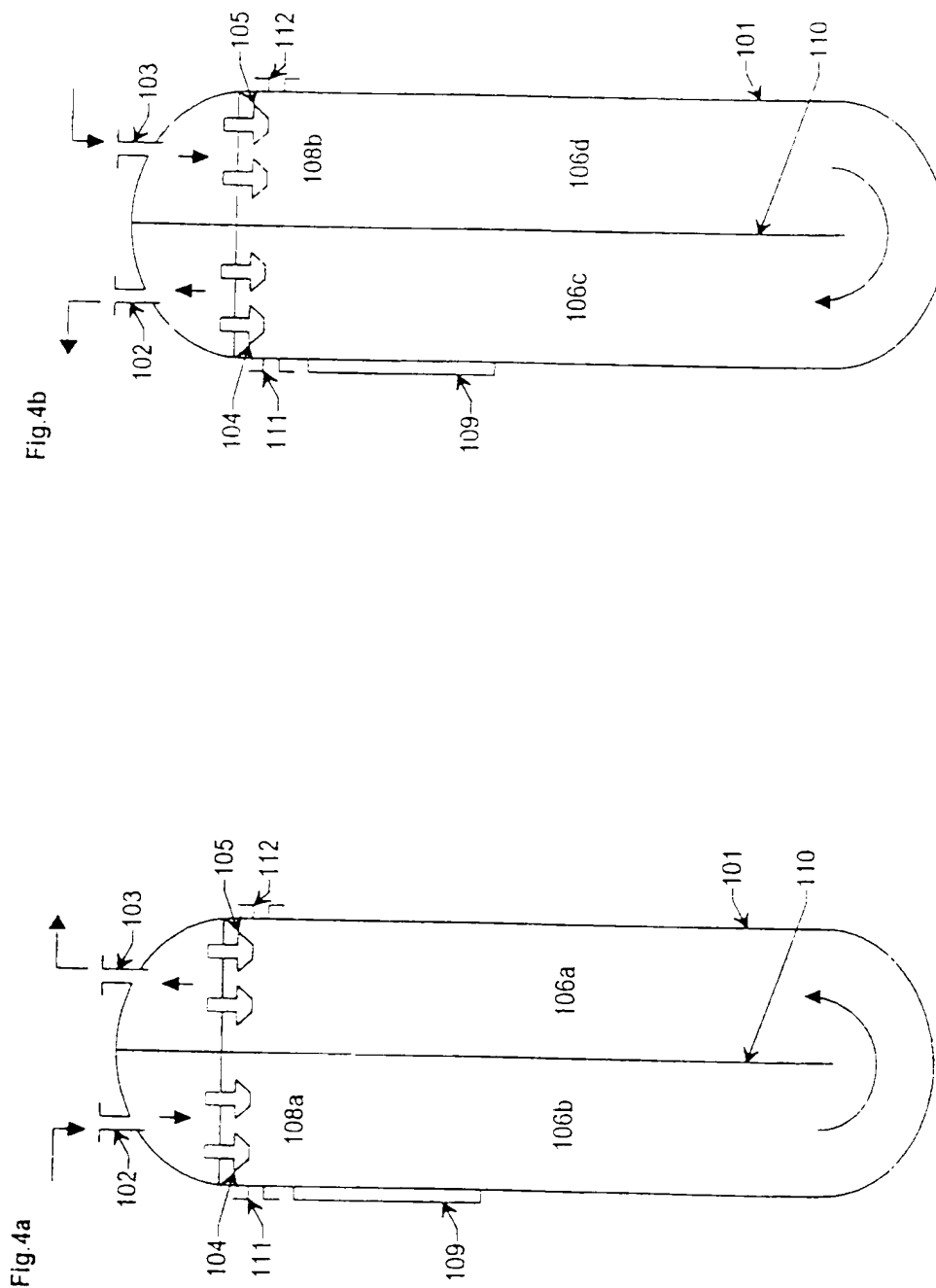


Fig. 3b





5/12

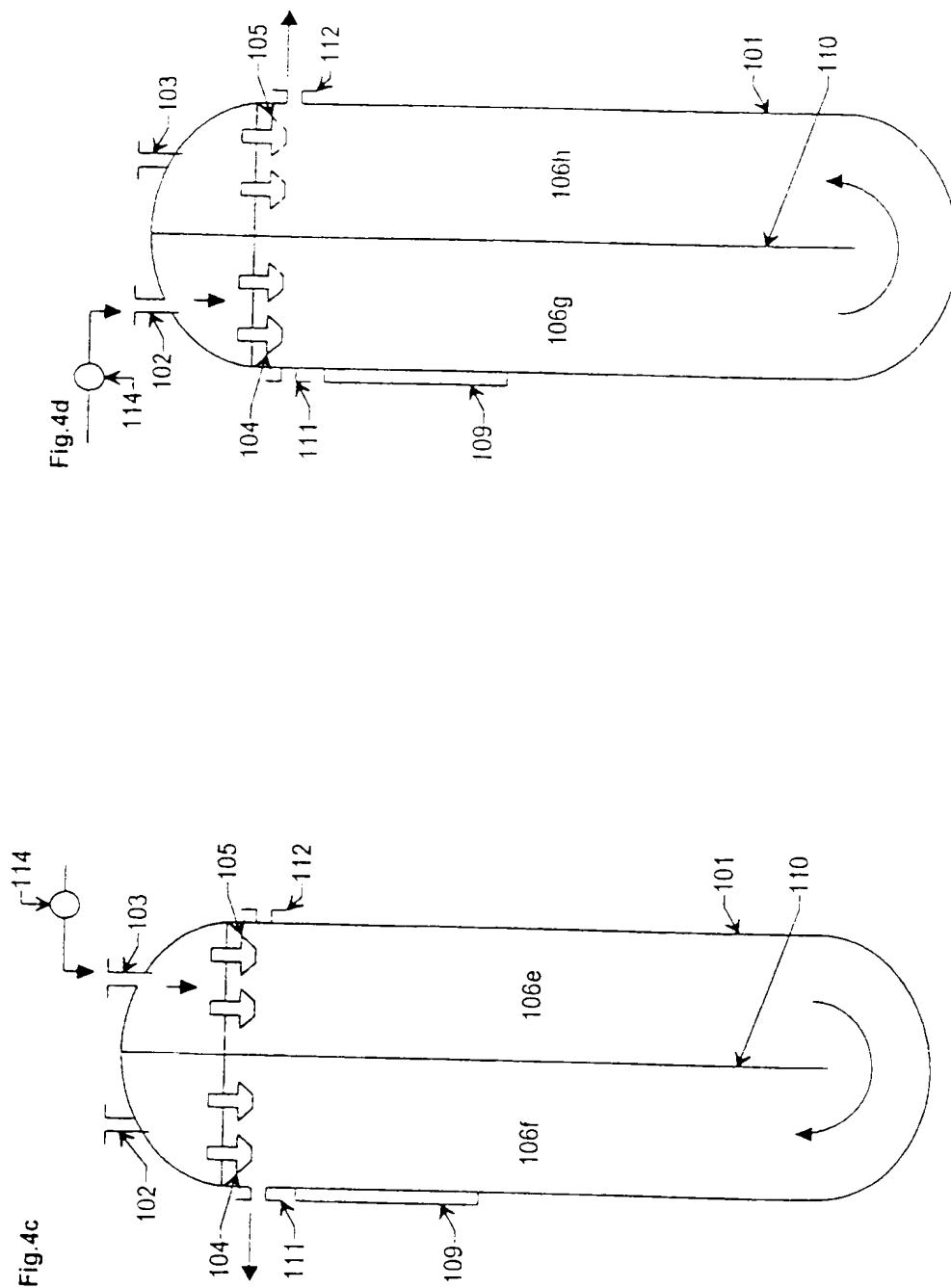
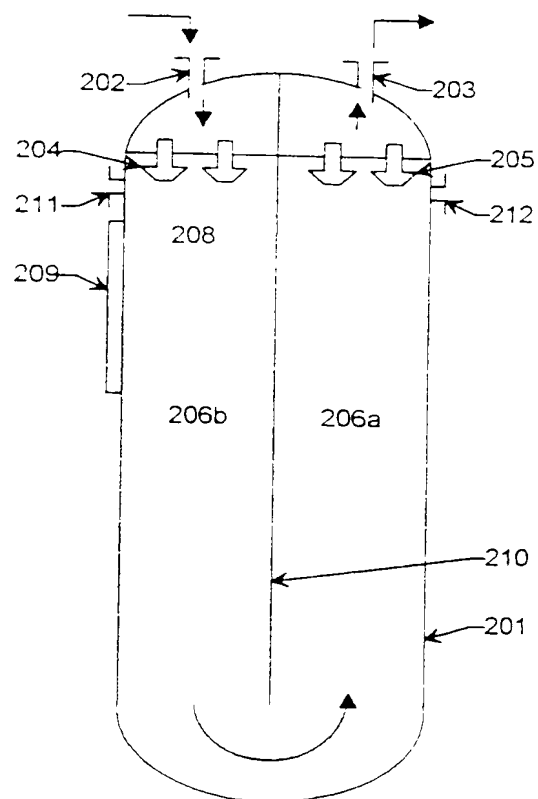
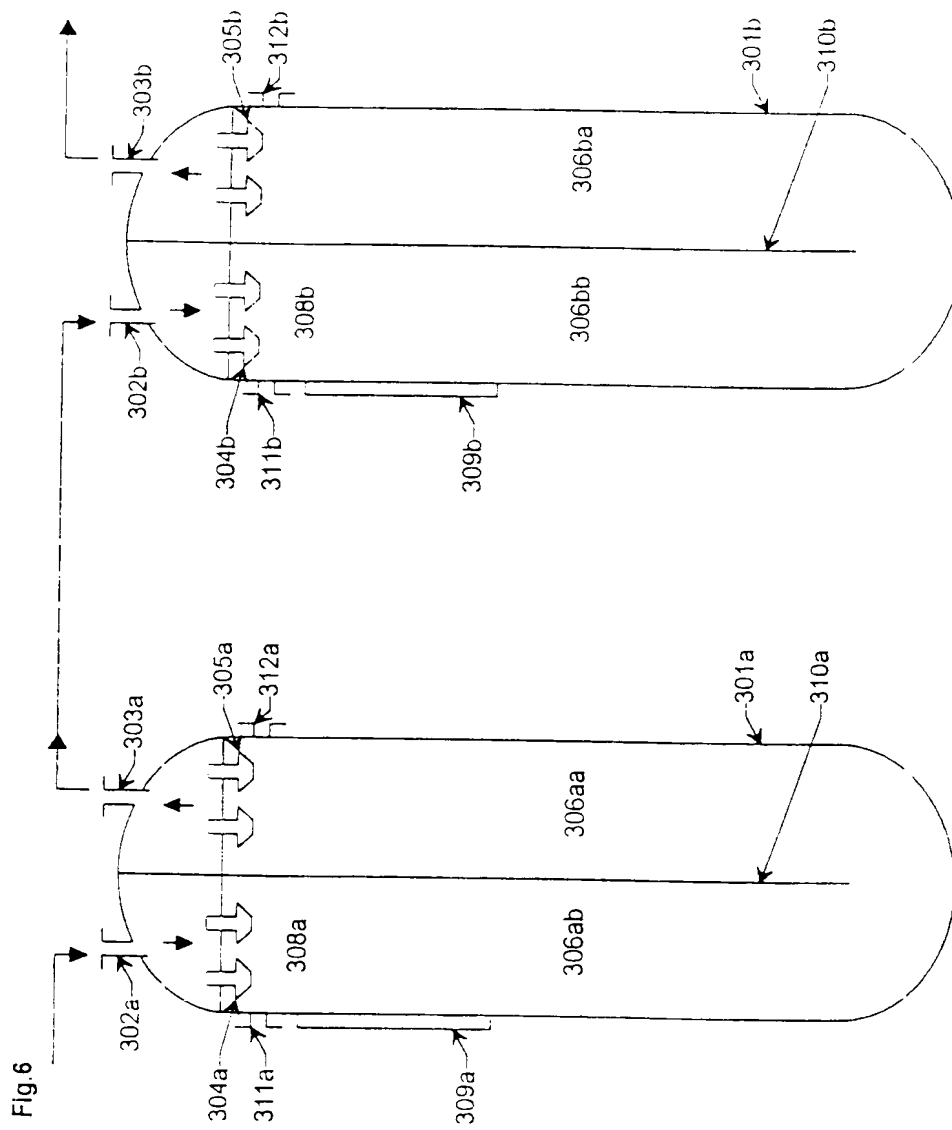
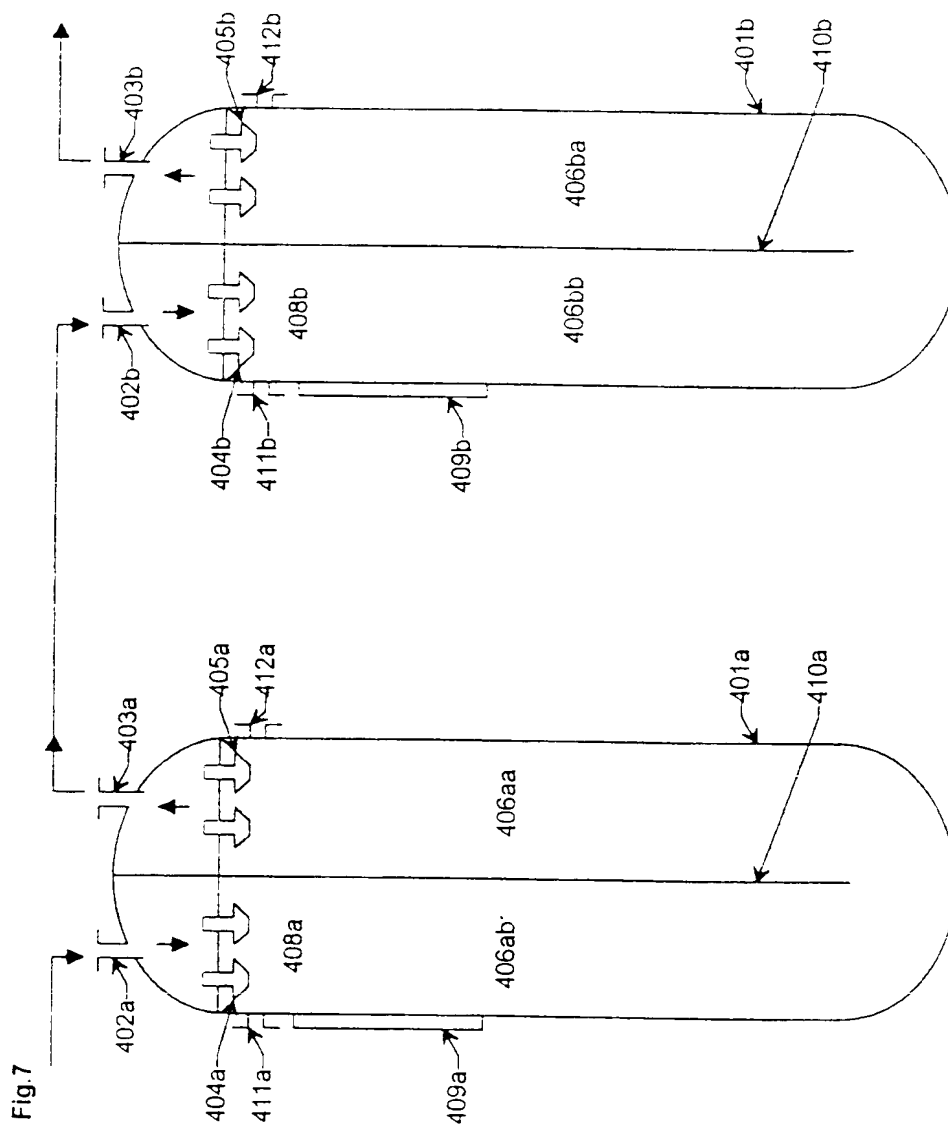


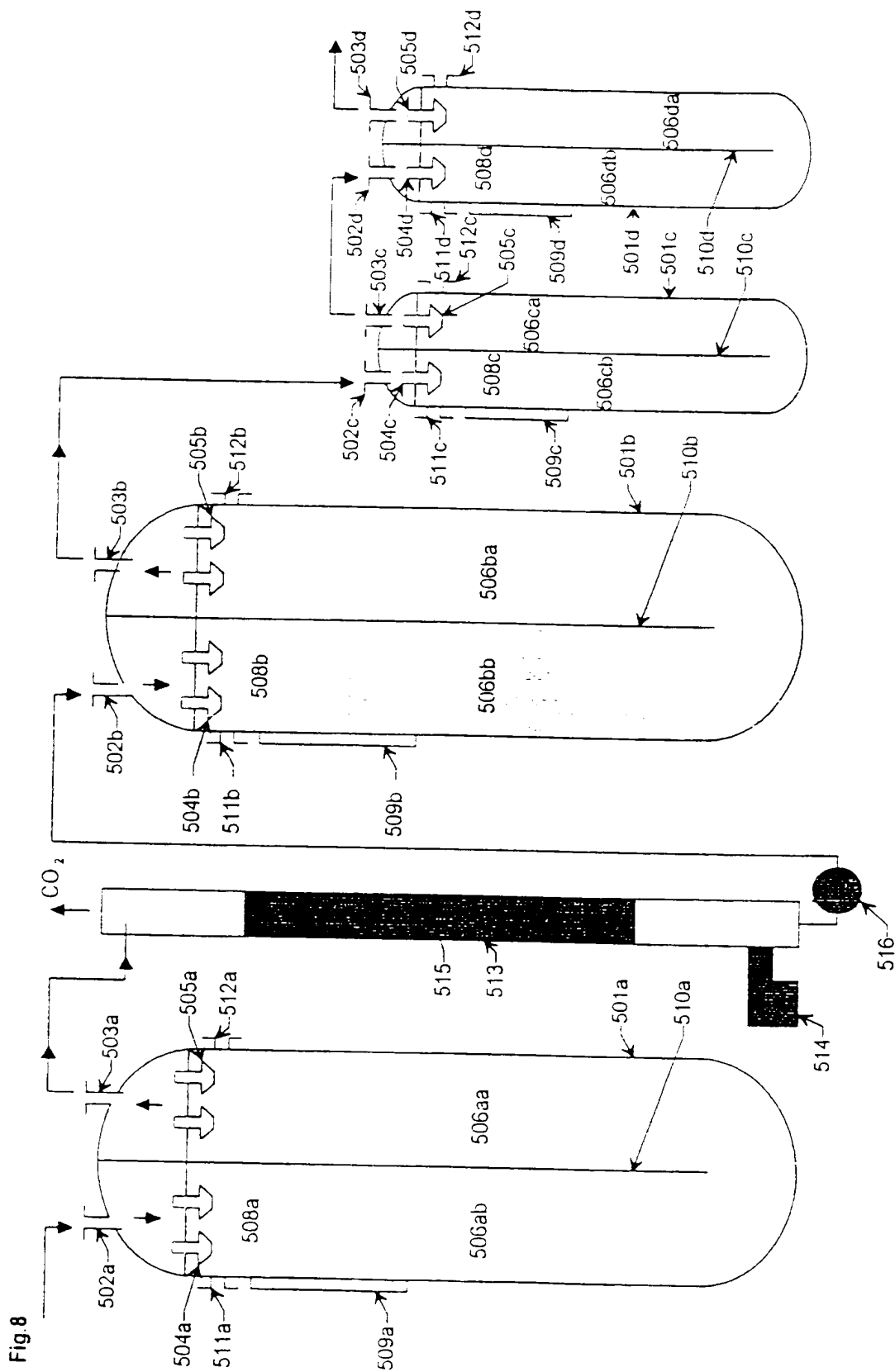
Fig.5





8/12





10/12

Fig.9a

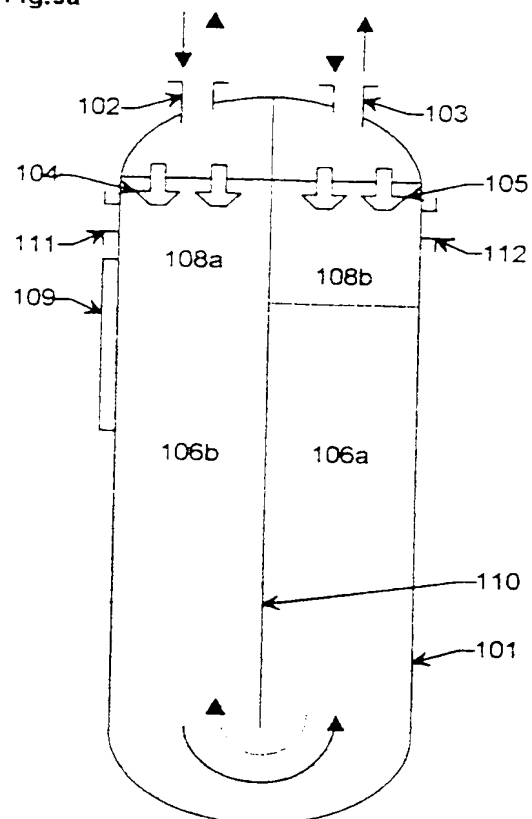


Fig.9b

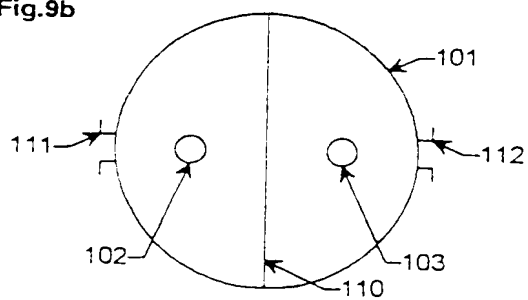


Fig.10a

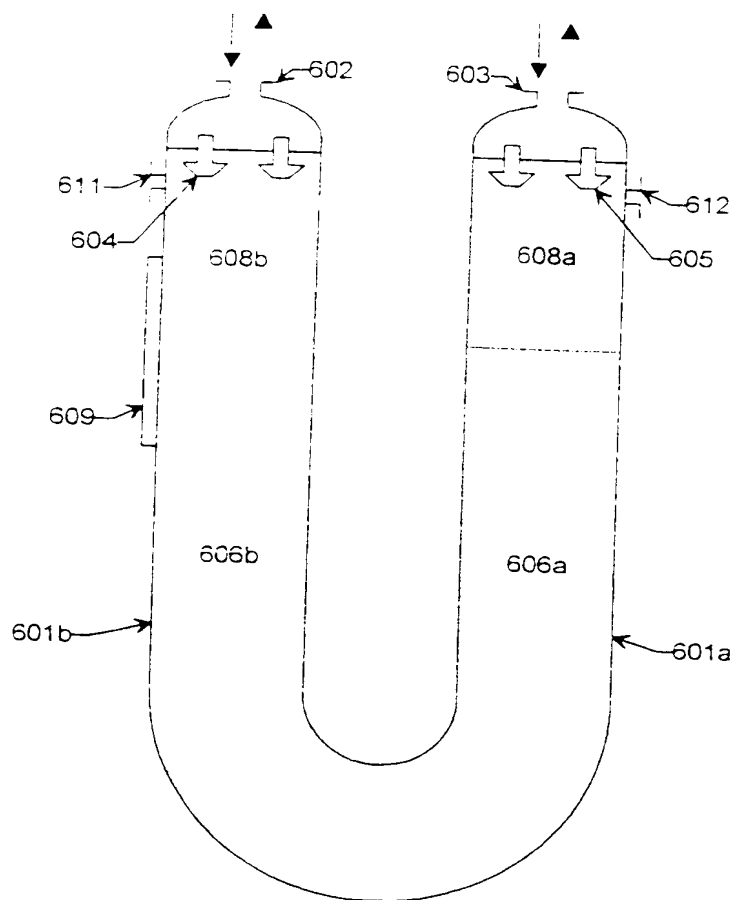
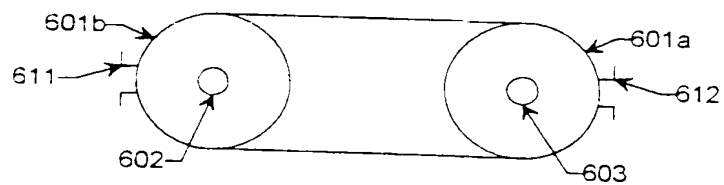


Fig.10b



12/12

Fig.11a

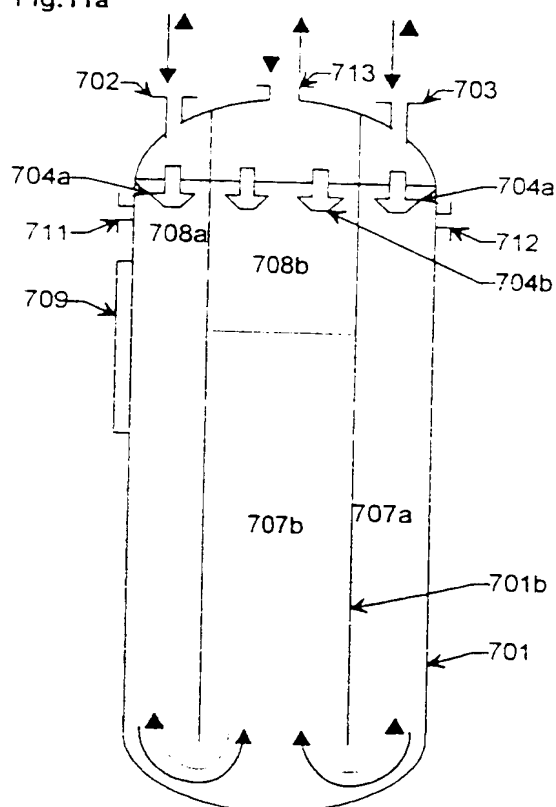


Fig.11b

